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Investigation in Applications of Hydrolyzed Industrial Activated Sludge: Operation of Unit BX, Kingsport, TN

Jacob E. Hutton
University of Tennessee - Knoxville

To the Graduate Council:

I am submitting herewith a thesis written by Jacob E. Hutton entitled "Investigation in Applications of Hydrolyzed Industrial Activated Sludge: Operation of Unit BX, Kingsport, TN." I have examined the final electronic copy of this thesis for form and content and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science, with a major in Chemical Engineering.

Paul R. Bienkowski, Major Professor

We have read this thesis and recommend its acceptance:

Robert M. Counce, Paul D. Frymier

Accepted for the Council:
Dixie L. Thompson

Vice Provost and Dean of the Graduate School

(Original signatures are on file with official student records.)

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Investigation in Applications of Hydrolyzed Industrial Activated Sludge: Operation of Unit BX, Kingsport, TN

A Thesis
Presented for the
Master of Science Degree
The University of Tennessee, Knoxville

Jacob Eli Hutton
May 2007

DEDICATION

This thesis is dedicated to my father Robert E. Hutton, my mother Elaine M. Hutton, my step-mother Patricia C. Hutton, my brother Robert E. Hutton II, and my aunts, uncles, and cousins for encouraging me to reach further in my education to achieve my goals. Additionally, I dedicate this work to: my close friends Aaron Brummit and Ed Hicks; advisors Paul Bienkowski, Robert "Pete" Counce; Eastman colleagues C. Calvert Churn III and John Barber; Rauschert Process Technologies, Inc. colleagues Daniel Mock, Chris Castleberry, Kari Lopez, Christine McKernan, Ahmed Elalfy; and, Jackie Bates for reinforcing my interest in completing this project.

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ABSTRACT

"Investigation of Hydrolyzed Sludge: Operation of Unit BX, Kingsport, TN" examines the treating of 15 weight percent activated sludge in a 1 ton per day pilot plant, Unit BX, with a lysing agent and heat to release the bound water contained in the activated sludge. The end-results were an assessment of the challenges for processing 15 weight percent sludge and collection of a product with water-like flow properties that is nutrient-rich suitable for the consumption by microorganisms (hydrolyzate). In addition to the development and execution of this pilot plant and examination of the functional properties of the hydrolyzate(total solids, viscosity, TOC, and BOD_5), two studies were conducted with either the properties of the experimental samples or with the samples collected (from Unit BX) . These feasibility studies are recycling the hydrolyzate to a wastewater treatment (WWT) facility for reducing the hydrolyzate to carbon dioxide or slurring the hydrolyzate with coal to assess the functional properties of this mixture for potential application to a commercial gasification process.

For the evaluation of recycle of hydrolyzate to WWT, a model was developed to project the impact of recycling the hydrolyzate to WWT, Plate 1, using the property data of the hydrolyzates. Results indicate that low concentrations of lysing agent appear to be attractive; however, the presence of inerts in the WWT influent would accumulate in the recycle making this option unfeasible.

For the evaluation of slurrying hydrolyzate with coal, coal grinds with the hydrolyzate were made to compare the functional properties of a coal grind with hydrolyzate (total solids, pH, viscosity, and coal particle size distribution) with the properties of a coal grind that does not contain hydrolyzate. Findings show that at low

temperature and low lysing agent or with an acidic lysing agent yield viscous and or foamy coal grinds. However, hydrolyzate samples collected at 150ºC with a basic lysing agent yielded physical properties that were comparable to a coal grind without hydrolyzate. In addition, the basic hydrolyzate showed promise as an effective agent for suspending coal particles in water compared to the standard coal grind and possible substitution with other additives.

PREFACE

This research is a phase 1 analysis of taking an organic solid waste stream (activated sludge) from an industrial Wastewater Treatment (WWT) facility and hydrolyzing in a process using a lysing agent. The objectives of this research are: to assess the challenges of operation of a hydrolyzate process; determine the different qualities of the treated hydrolyzate from this process; evaluate the feasibility of recycling treated hydrolyzate back to WWT for consumption by microorganisms to create harmless carbon dioxide using a model developed (Plate 1); and, determine if the hydrolyzate can slurry with coal for a commercial gasification process. Overall, this research is to evaluate the feasibility of two pathways to achieve zero solid waste emissions from a industrial WWT facility.

During the research period between May 2004 and August 2004, a 1 ton per day catalytic hydrolysis process for 15 weight percent activated biosludge (Unit BX) was constructed and operated at Eastman Chemical Company, Kingsport, TN. The process was a success with challenges. Continuous operation was maintained for a period of 62 hours. 18 experiments were performed, 16 experiments using sodium hydroxide and 2 for sulfuric acid. A total of 44 two (2) quart samples of sodium hydroxide and 5 two (2) quart samples sulfuric acid treated hydrolyzate were collected. Three sources for system instability were identified. These sources are: bridging of biosludge in the feed hopper, erratic flow control of the treated hydrolyzate, and unsteady state heating of the system. Primary strategies to maximize operation performance and minimize the impact of any of these sources for instability are: preventing bridging in the feed hopper, using a valve configuration to manage the non-Newtonian characteristics of the hydrolyzate, and

steam-jacketing the process lines to the inlet of the continuous stirred tank reactor. The primary variable that could shutdown the operation of Unit BX was pressure either from gas collecting in the reactor from the sludge stream, overheating biosludge/lysing agent mixture and hydrolyzate in the system, gas generation from a neutralization reaction, or overfilling the system. Results indicate that operation of a catalytic hydrolysis process has a stable operation at total flow rates approx 600 $gmin^{-1}$, a residence time of approximately 10 minutes, reactor pressures greater than 80 psig and less than 120 psig, and at temperatures greater than 140ºC and less than 170ºC.

Results from Plate 1 show that low concentrations of sodium hydroxide and sulfuric acid used to treat biosludge have potential for return to WWT. In contrast, the presence of inerts in the influent stream to WWT has a dramatic impact on the flow rate of the recycle stream at greater amounts of biosludge recycled, making this option uneconomical.

Coal slurry tests using a mixture of water and treated hydrolyzate were performed at the University of Tennessee-Knoxville. Hydrolyzate/water mixture slurries that had poor performance were the sodium hydroxide treated hydroxide samples that were heated to 140ºC and low lysing agent concentrations and sulfuric acid treated hydrolyzate slurries. The poor performance was due either the viscous nature of the slurry for the sodium hydroxide hydrolyzate at low concentrations of lysing agent and the foaming tendencies of the sulfuric acid hydrolyzate slurries (viscosities greater than 40,000 cP). In contrast, results indicated that it is possible to slurry coal with the sodium 150ºC hydroxide treated hydrolyzate. The slurries from this treated hydrolyzate temperature range had a viscosity 8,000-12,000 cP (approximately 2-3 times the viscosity of the

standard coal slurries) and an average particle distribution of solids comparable to the standard coal slurry. Results from the coal slurries in general shown that the suspension properties of the coal solids in water were enhanced by the addition of the sodium hydroxide treated hydrolyzate and that the use of a surfactant (ammonium lignin sulfate (ALS)) could be left out of the coal slurry mixture.

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NOMENCLATURE

F_{LA}	Mass flow rate of lysing agent ($g \cdot min^{-1}$, $lb \cdot day^{-1}$)
F_{WS}	Mass flow rate of wet sludge ($g \cdot min^{-1}$, $lb \cdot day^{-1}$)
F_{LA}	Mass flow rate of dry sludge ($g \cdot min^{-1}$, $lb \cdot day^{-1}$)
C_{LA}	Concentration of lysing agent ($g\text{-}mol \cdot L^{-1}$, $lb\text{-}mol \cdot gal^{-1}$)
MW_{LA}	Molecular weight of lysing agent (g-mol, lb-mol)
K_{LA}	Proportionality constant between lysing agent flow rate and wet sludge flow rate
μ_{DBLA}	Design basis specific to lysing agent ($lb_{LA} \cdot lb_{DS}^{-1}$)
D_B	Design basis multiplier (%)
F_x	Mass flow rate of stream x or specie x ($g \cdot min^{-1}$, $lb \cdot day^{-1}$)
F_{x-y}	Mass flow rate of specie y in stream x ($g \cdot min^{-1}$, $lb \cdot day^{-1}$)
$\dot{V}_x$	Volumetric flow rate of stream x or specie x ($mL \cdot min^{-1}$,$gal \cdot day^{-1}$)
$\dot{V}_{x-y}$	Volumetric flow rate of specie y in stream x ($mL \cdot min^{-1}$,$gal \cdot day^{-1}$)
ρ_x	Density of stream x ($g \cdot L^{-1}$, $lb \cdot gal^{-1}$)
γ_{conv}	Unit conversion factor ($0.012016\ lb \cdot L \cdot min \cdot mg^{-1} \cdot gal^{-1} \cdot day^{-1}$)
θ	Percent dry biosludge per wet biosludge ($lb_{DS} \cdot lb_{WS}^{-1}$)
θ_x	Percent specie x per wet biosludge (($lb_x \cdot lb_{WS}^{-1}$)
χ	Percent sludge sent away from WWT (%)
$1-\chi$	Percent sludge sent to recycle (%)
Γ	Treated sludge substitution factor (gal·lb)
η_{Carbon}	Percent carbon in carbon dioxide (%)
λ_{Carbon}	Percent carbon in biomass (%)
λ_{oxygen}	Percent oxygen in biomass (%)
β_x	Percent inerts in biosludge (%)
$\nu_{\frac{BOD_U}{BOD_5}}$	BOD_U to BOD_5 ratio (1 lb BOD_U per 0.7 lb BOD_5)
$\Delta_s Y$	Biomass yield per pound BOD_U (0.42 lb per 1 lb BOD_U)
ψ	Biomass yield, BOD_U to BOD_5 ratio, and unit conversion factor for substitution and equation simplification)
min	Minute
mg	Milligram
g	Grams
lb	Pound
L	Liter
gal	Gallon

g-mol	Gram moles
lb-mol	Pound moles
$g \cdot min^{-1}$	Grams per minute
$lb \cdot day^{-1}$	Pounds per day
$g \cdot g\text{-}mol^{-1}$	Grams per gram mole
$g\text{-}mol \cdot min^{-1}$	Gram moles per minute
$g \cdot mL^{-1}$	Grams per minute
$g\text{-}mol \cdot L^{-1}$	Gram moles per liter
$L \cdot min^{-1}$	Liters per minute
$g_{LA} \cdot g_{WS}^{-1}$	Grams of lysing agent per gram of wet sludge
$g_{LA} \cdot g_{DS}^{-1}$	Grams of lysing agent per gram of dry sludge

ABBREVIATIONS

TOC	Total Organic Carbon ($mg \cdot L^{-1}$)
TOC_x	Total Organic Carbon of stream x ($mg \cdot L^{-1}$)
TIC	Total Inorganic Carbon ($mg \cdot L^{-1}$)
TC	Total Carbon ($mg \cdot L^{-1}$)
BOD_5	Biochemical Oxygen Demand 5 day test ($mg \cdot L^{-1}$)
$BOD_{5\text{-}x}$	Biochemical Oxygen Demand 5 day test of stream x ($mg \cdot L^{-1}$)
BOD_U	Biochemical Oxygen Demand Ultimate test
I	Inerts Concentration ($mg \cdot L^{-1}$)
WWT	Waste Water Treatment
WAO	Wet Air Oxidation
LPO	Low Pressure Oxidation
LA	Lysing Agent
WS	Wet Sludge
CO	Carbon monoxide
CO_2	Carbon dioxide
NaOH	Sodium hydroxide
H_2SO_4	Sulfuric acid
H_2O	Water
ALS	Ammonium lignin sulfate
$C_5H_7O_2N$	Empirical formula for microorganisms

CHAPTER 1 INTRODUCTION

BACKGROUND INFORMATION

In the United States approximately 5.6 million dry tons of biosludge is generated annually from wastewater treatment processes [1]. A significant portion of this sludge comes from industrial processes. After a primary dewatering of this industrial sludge on a belt filter press, a solid-like material is produced with a solids concentration of 12 – 20 weight percent [2]. The solids are primarily dead biomass and represent a disposal problem and a operating cost. Examples of some disposal methods include land farming, incineration, and wet air oxidation [3, 4, 5]. A primary processing problem is the bound water within the biomass, which results in very high fluid viscosities and requires large inputs of energy during the disposal process. A catalytic hydrolysis process has been developed which releases the bound water, resulting in a low viscosity stream rich in soluble and suspended organics suitable for recycle to a wastewater treatment facility. The organic compounds in this stream represent a significant energy source which could be recovered by slurrying with coal in a coal gasification process.

The objective of this research project is to evaluate the feasibility of operating a scaled up continuous pilot process using 15 weight percent viscous biosludge, and evaluating the potential of returning lysed biosludge to wastewater treatment (WWT). A mass balance model will be used to evaluate the effects of recycling this material to WWT, coal slurry samples will be produced from the effluent stream of this pilot plant to evaluate the feasibility of utilizing lysed biosludge as a feed material to a coal gasification process. Key results indicate that operation of a catalytic hydrolysis process

has a stable operation at total flow rates approx 600 $gmin^{-1}$, a residence time of 10 minutes, reactor pressures greater than 80 psig and less than 120 psig, and at temperatures greater than 140ºC. Additional findings show that low concentrations of sodium hydroxide and sulfuric acid used to treat biosludge has potential for return to WWT, and that elevated concentrations of sodium hydroxide and hydrolysis temperatures at approximately 150ºC have potential to be adapted to coal slurry. Research methodology will include: introduction of examples of current methods of biosolids disposal for comparison; literature review of other methods for lysing biosolids disposal operation; operation of a catalytic hydrolysis process, Unit BX; evaluation of the recycle of treated hydrolyzate to WWT; and, adaptation of biosolids hydrolyzate to form coal slurry.

1.2 EXAMPLES OF CURRENT DISPOSAL METHODS OF BIOSOLIDS

In a climate of escalating environmental regulations and restrictions regarding where and how to dispose of the biosludge, the hardships involved translate to escalating costs. Current practices used for biosolids disposal include land farming, incineration, and wet air oxidation (WAO).

Land farming is a practice where the biosolids are applied directly to land and allowed to degrade naturally. The Knoxville Utilities Board (KUB) uses this method. KUB ships their biosolid wastes more than 70 miles from Knoxville, TN to be land farmed [6]. The same farm land can not be used year after year and the process must be rotated. This method is coming under more scrutiny due to how biosolids generation will continue to increase with increasing populations, rising costs of disposal and inflating transportation costs.

Incineration is a practice where the biosolids are completely oxidized to CO_2, leaving ash and any noncombustible materials for collection and offsite disposal [2]. For example, Eastman Chemical Company in Kingsport, TN, burns their solid 15 weight percent biomass in their onsite boilers to eliminate this waste stream [7]. Some realities of this process are that environmental regulations continue to become more stringent on how the biosolid wastes are disposed of. For instance, Eastman was cited for an environmental monitoring penalty due to a malfunctioning mass flow meter in the measurement of how much biosolid material was being disposed of over a particular period of time[8].

Wet air oxidation (WAO) is a process that uses high temperatures and oxygen from air to oxidize difficult wastewater streams [2, 5]. The main feature of this process is that the organics and the oxygen combine to break down the organics into low molecular weight acids, carbon dioxide and water. Another process that is available is low pressure oxidation (LPO) where temperatures and pressures are less than traditional WAO, but still achieve oxidation. In some circumstances, this method has been demonstrated to reduce the solids concentration of the activated sludge. In contrast, this process has inherent operating challenges due to high maintenance and energy costs [2].

1.3 LITERATURE REVIEW OF OTHER METHODS FOR LYSING BIOSOLIDS

There are studies that promote waste reduction in processes using a lysing agent in conjunction with thermal treatment. Frequently tested lysing agents tested are nitric acid, sulfuric acid, and sodium hydroxide. Two agents of particular interest are sulfuric

acid and sodium hydroxide due to low cost and/or excellent material compatibility, respectively.

The use of nitric acid is mentioned in works by Perkins et. al. [9, 10, 11] This application is also referred to as nitrolysis. The basis for nitrolysis is to use activated sludge and mix it with portions of nitric acid in a heated system to lyse the biosolids and decompose the macromolecules to acetic acid as a product. The key finding of Perkins work are that temperature, the acid to sludge ratio, and time are the main factors that inpact converting the biomass into a water-like material. Additionally, lower concentrations of acid and increased sludge solids content up to 15 weight percent sludge were used to show that similar effects in converting the biomass into a water-like material. Conceivably, small amounts of lysing agent could be used to lyse a high solids content biomass and release the bound water with thermal treatment thus improving the handling and dewatering characteristics of the biosludge. An additional example of nitrolysis is a solids waste reduction regimen presented in patent documentation by William Schotte. The basic process is the reduction of the pH using nitric acid while performing wet air oxidation. The results demonstrate that filterability of the biosludge is increased resulting in an increased filter cake solids content [12].

An example of hot acid hydrolysis is presented by Neyens et. al. [13] This article shows that the application of sulfuric acid with thermal treatment demonstrates similar effects compared to nitrolysis by means of allowing further dewatering of the biosludge along with other potential applications of material and energy recovery.

Hydrolysis of biosludge using sodium hydroxide has been indicated in patent documentation for waste reduction. An example is patent documentation by Aale

Pasveer, which shows that hydrolysis of the biosludge using sodium hydroxide is a viable alternative for waste reduction [14].

There are many works supporting the addition of a lysing agent to adjust the pH coupled with an increased energy input, resulting in easier management of biosolids. However, few publications report the challenges to processing a high solids content sludge, what equipment to use, or the potential for energy recovery. In a presentation made at the American Institute of Chemical Engineers conference 2004 in Austin, TX, Hutton et. al. [15] presented a topical discussion of investigations relating to the costing of different alternatives providing strong incentives for two catalytic lysing agents for certain operational properties: sodium hydroxide and sulfuric acid. This presentation provided a comparison of potential operation of a batch and continuous system using different lysing agents and compared the capital and operational investments of each lysing agent for the application to a wastewater treatment alternative. Two potential lysing agents, sulfuric acid and sodium hydroxide, were deemed viable due to either the reasonable cost of the lysing agent or the operating unit's material of construction. From this evaluation, the operation of Unit BX using either sodium hydroxide or sulfuric acid as a lysing agent was considered.

1.4 Design Basis

A design basis is a variable used in all experiments. One design basis or 100% design basis for sodium hydroxide denotes 0.70 pounds of 50 weight percent sodium hydroxide per pound of dry sludge. For sulfuric acid, one design basis or 100% design basis is 0.35 pounds of 98 weight percent sulfuric acid per pound of dry sludge [16].

This unit of measure extends from earlier economic studies that were established for application of the treated biosludge to wastewater treatment (WWT) or adaptation of the treated biosludge to coal grinds for gasification. In addition, the total molar electric charge for each lysing agent with respect to the amount of dry sludge is equal. This will allow equal comparisons between the lysing agents.

The experimental design for sodium hydroxide includes multiples of 200%, 100%, 75%, and 50% of the design basis used to evaluate potential the range application of biosludge for application to wastewater treatment and adaptation of the treated biosludge to coal grinds for gasification. For sulfuric acid, multiples of 100% and 50% of the design basis are used for testing.

The lower multiple of the design basis for sodium hydroxide is the bottom range for recycle to WWT for the case that the economics of the process fit to allow for enough credits from incineration reduction and credits from the reduction of calcium hydroxide usage in WWT for the sodium hydroxide case only. The higher multiple of the design basis for sodium hydroxide is the top range for adapting the application of biosolids to gasification for the case that the economics of the process fit to allow for credit from incineration. In addition, the expectation is that the higher concentration of lysing agent would increase the soluble organic compounds, increasing the energy content in the liquid volume of the coal sludge slurry.

CHAPTER 2 OPERATION OF UNIT BX

2.1 OPERATION OF A CATALYTIC HYDROLYSIS PROCESS, UNIT BX

Unit BX is a one (1) ton per day demonstration unit specifically designed to load the cake-like consistency fifteen (15) weight percent activated sludge into a heated pipe network feeding to a continuous stirred tank reactor with a lysing agent, sodium hydroxide or sulfuric acid. The mixture is heated to the desired temperature and contained for an average residence time of 10 minutes in the reactor. Then the product is cooled, discharged, and samples can be taken.

The purpose of Unit BX is to perform studies for operating a system with fifteen weight percent activated sludge with a lysing agent, to obtain substantial product sample quantities (greater than two (2) liters) for further testing, to assess operational difficulties, and to find optimal operating conditions.

Topics discussed in the following sections include: a process description; process input variables; controlled variables of the system; measured process variables; tests to be performed on the product; and, anticipated experimental testing process conditions.

2.2 PROCESS DESCRIPTION

Unit BX consists of seven operating sections: the biosludge loading and feeding section; the lysing agent loading and feeding section; the biosludge-lysing agent mixing and pre-heating section; the lysis reaction/blending section; the cooling section; the fluid flow control section; and, the entire system feedback and control system. Figure 1 outlines the primary sections of Unit BX and the instruments used to take measurements of the whole process. Table 1 outlines the equipment and instrument identifications.

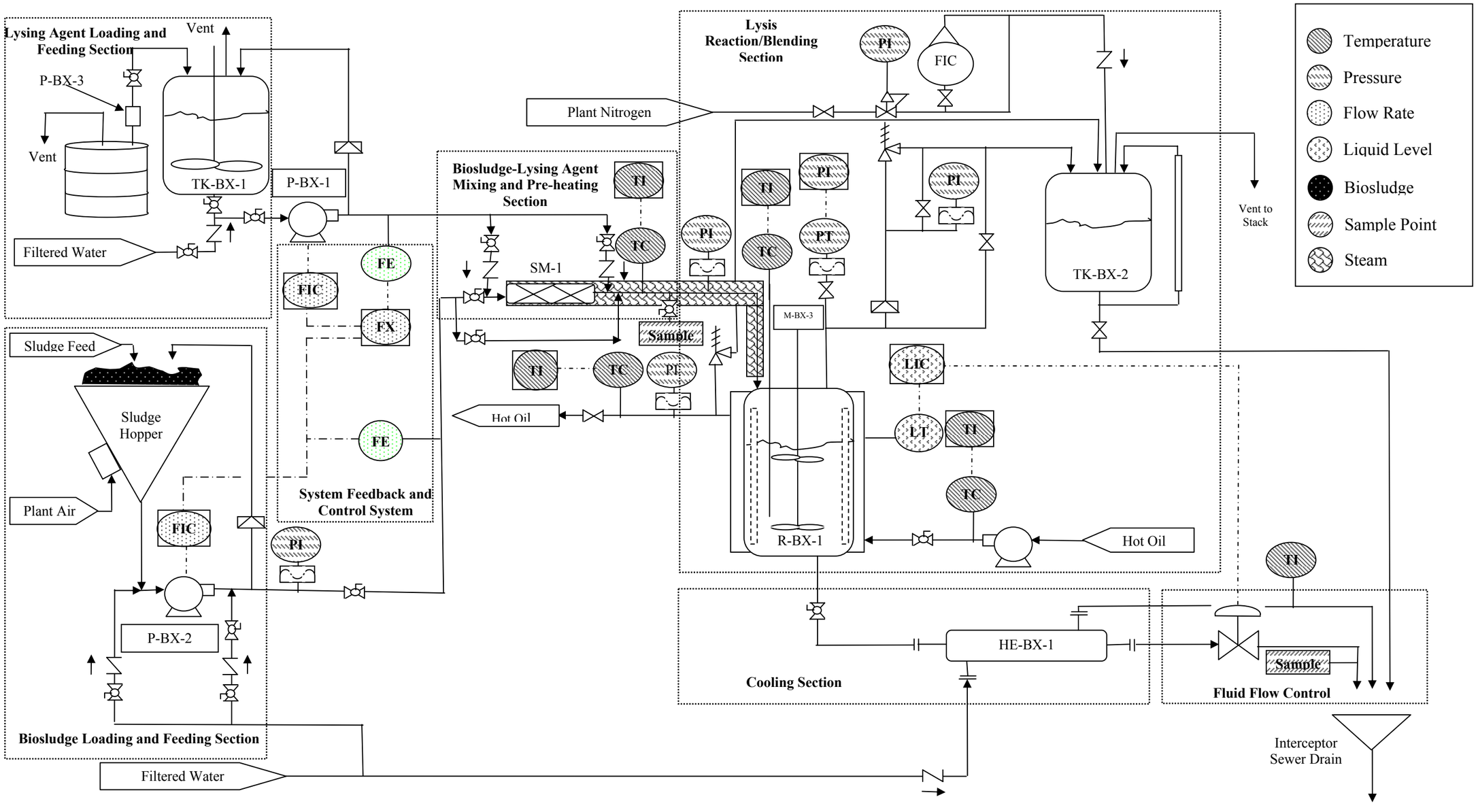
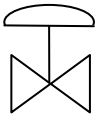


FIGURE 1 Process Flow Sheet of Unit BX

TABLE 1 Equipment Identification Table for Figure 1

General Description	Identification Tag or Image	Description
Pumps	P-BX-1	Milton Roy Chemical Injection Pump
	P-BX-2	Moyno Progressive Cavity Positive Displacement Pump
	P-BX-3	Pneumatic Drum Pump Wand
Tanks	TK-BX-1	Lysing Agent Storage/Feed Tank
	TK-BX-2	Emergency Relief Tank
Reactor	R-BX-1	Hot Oil Jacketed Continuous Stirred Tank Reactor
Static Mixer	SM-1	18 Element Static Mixer
Heat Exchanger	HE-BX-1	Water Cooled Heat Exchanger
Agitator	M-BX-3	Dual Impeller Magneto Drive Agitator
Valves		Ball Valve
		Check Vale
		Needle Valve
		Control Valve
		Pressure Relief Valve
Miscellaneous Equipment		Rupture Disc
		Instrument Isolation Grid Block
Connections		Fluid Flow Direction
		Piping Connection
		Electrical Signal Connection
Process Variable Instruments and Transducers	PI	Pressure Indicator
	TI	Temperature Indicator
	PT	Pressure Transducer
	TC	Thermocouple
	FE	Flow Meter
	LT	Level Transmitter
Process Controllers	FIC	Pump Flow Controller
	FX	Control System Flow Controller
	LIC	Liquid Level Controller

In the biosludge loading section, the sludge is loaded in the feed hopper from a stainless steel transfer hopper as shown in Figures 2 and 3. The stainless steel transfer hoppers filled with biosludge are lifted by a fork truck and manually unloaded into the feed hopper. After the sludge is loaded, it is fed into the system via an auger at the bottom of the feed-hopper in the Moyno pump transfer case. Additional aids to assist feeding the biosludge into the Unit BX include steel cladding of the inside surface of the feed hopper, a water jet to break up bridging, and air-hammer bin-vibrators.

Figure 4 shows the feed hopper and the Moyno pump. The Moyno pump is situated below the inverted pyramid shaped cone structure of the feed hopper.

FIGURE 2 15 Weight Percent Sludge in Transfer Hopper

FIGURE 3 Transfer Hoppers for Moving Sludge from Wastewater Treatment to Demonstration Unit

FIGURE 4 Sludge Feed Hopper Above Moyno Pump

Figure 5 shows the interior of the feed hopper. At the bottom of the feed hopper there is an auger that feeds the biosludge into the Moyno pump. Figure 6 shows a side view of the Moyno pump and how it connects the feed hopper to Unit BX. There is an over flow pipe shown that is used to maintain a steady liquid level in the Moyno pump transfer case and prevent over flow of the feed hopper. In addition, the overflow pipe allows for sludge removal with water obviate the need for a person to enter the interior of the feed hopper during system cleanout procedures. Figure 7 illustrates one of the two bin vibrators of the system to minimize bridging. In addition, the hose above the bin vibrator is a water supply connection used for flushing the feed hopper after experiment termination and keeping the Moyno pump wet during hot-standby operations.

After the sludge is fed into Unit BX, it enters the second section of the Unit BX, passes through a Coriolis mass flow meter where the pump speed can be adjusted to maintain the desired flow rate of the system shown in Figure 8.

After the mass flow meter, the sludge is fed into a steam heated portion of the system, where the biosludge is mixed with the choice of lysing agent in an 18 element static mixer shown in Figures 9 and 10. Figure 9 is a view from the top of the static mixer looking down. Figure 10 is a side view of the static mixer. As seen in figure 10 the static mixer is steam-jacketed and insulated. The static mixer bypass is the uninsulated pipe to the left in the photograph. Figure 11 illustrates the lysing agent storage and pumping utilities to feed lysing agent to the sludge stream. The lysing agent agent is fed into the storage tank from a 55 gallon barrel with a diastolic pump.

FIGURE 5 Inside of Feed Hopper, Clad with Stainless Steel to Facilitate Sludge Flow to Moyno Pump

FIGURE 6 Moyno Pump for Feeding Sludge to the Demonstration Unit Reactor

FIGURE 7 Mechanical Vibrator to Prevent Sludge Plugging in the Feed Hopper

FIGURE 8 Mass Flow Meter for Controlling Sludge Feed Rate

FIGURE 9 Static Mixer with steam Jacket

FIGURE 10 Plug Flow Reactor with Steam Jacketed Static Mixer

FIGURE 11 Catalyst Feed System with Metering Pump and Mass Flow Controller

The storage tank is sealed to prevent air mixing with the lysing agent. The lysing agent is fed into Unit BX through the chemical injection Milton Roy pump shown in the bottom left of the photograph. In the upper section of the photograph, a second coriolis mass flow meter measures the amount of lysing agent that is injected into the system. Mixing takes place by means of the flow of the sludge and the lysing agent through the static mixer reaching effective pre-mixing and pre-heating. The resultant material is held up in the continuous stirred tank reactor (CSTR) for a mean residence time of 10 minutes. Here the liquid level is maintained and the influent reaches the desired temperature.

The CSTR is shown in Figures 12 and 13. Figure 12 shows the top of the reactor and how it is insulated. In addition, Figure 12 illustrates the complex arrangement of relief valves, pressurization valves, the mechanical motor drive of the agitator, and part of a Stromand valve for taking reactor influent samples while the system is in operation

FIGURE 12 Sealed CSTR, Reactor Feed Sampling Port is in the Foreground Center

FIGURE 13 Inside View of the Open CSTR Reactor

under pressure. Figure 13 shows open CSTR with the Hastelloy® B-2 lining, 4 agitator baffles, and the bottom drain of the reactor. Figure 14 shows the open CSTR at the top flange with the agitator shaft and impellors. The agitator is a magneto drive agitator where the system is completely sealed and the agitator is driven by a set of magnets on the exterior of the vessel top. This is necessary to avoid any potential system seal issues. To the right of the agitator shaft is a thermowell to allow the use of a temperature measurement probe to monitor the mixture temperature. After a 10 minute residence time in the CSTR, the effluent (or treated biosludge hydrolyzate) is immediately cooled in a simple shell and tube heat exchanger. Shown in Figure 15, the product flows through the blow-case valve assembly, which is used to maintain system pressure, proper liquid flow rate, and an effective liquid level.

Figure 16 illustrates the discharge point from Unit BX. This is where samples of the hydrolyzate are collected for testing and excess hydrolyzate between sampling is sent to WWT sewer connection. To the lower right is a drum that sets on a scale. The drum was intended to be used for mass and energy balances on the system; however, the drum was not used during the research period covered by this document.

Figure 17 illustrates the control screen for Unit BX in the control room. It is a Human-Machine-Interface (HMI) to allow the operator to monitor the operation of the system, change operation variables, and forecast potential system upsets. In addition to this HMI, a historian (or computer data storage system) is also connected to the system to record properties of all measured process variables for later evaluation of system performance.

FIGURE 14 Open CSTR Reactor at Top Flange and Agitator Shaft with Impellors and Neighboring Thermowell

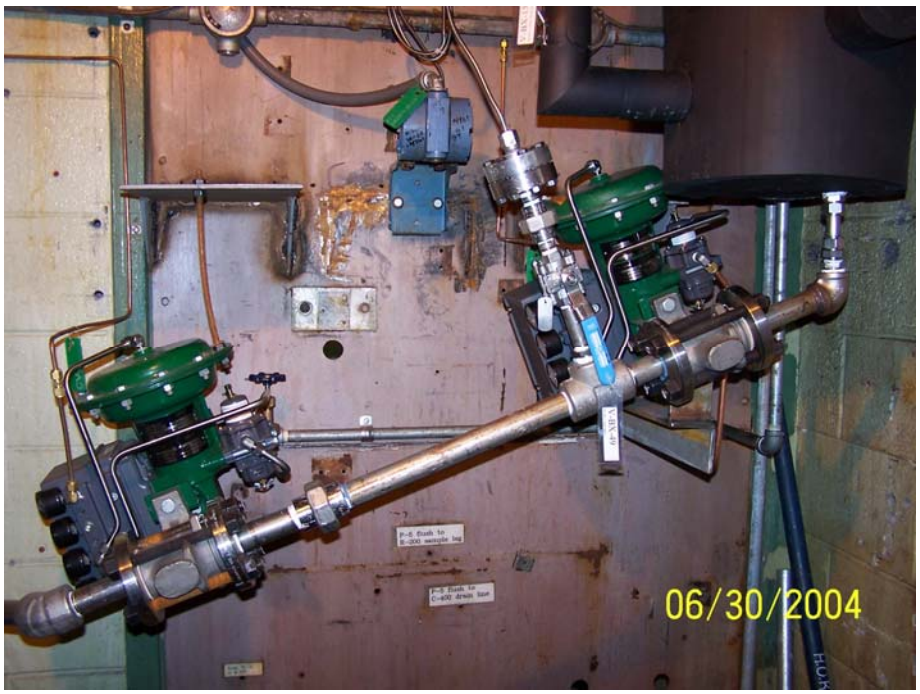


FIGURE 15 Control Valves on a 45 Degree at Outlet of Reactor Cooler

FIGURE 16 Product Sampling and Storage

FIGURE 17 Display Panel Taken from Unit Control Screen

2.3 PROCESS VARIABLES OF UNIT BX

As shown in section 2.2, Unit BX is a collection of systems or unit operations that are coordinated to achieve the goal of processing dense 15 weight percent biosludge with a lysing agent to yield a water-like energy-rich substance. Each of these subsections collectively operate together on sets of process variables to achieve the aforementioned outcome. These process variables are: process input variables and process manipulated variables/process controlled variables. These variables define the nature of the process. The post operation variables show the performance of the process.

2.3.1 Process Input Variables

Two primary variables that affect the processibility of biosludge are solids content and the microbiology of the biosludge. Process input variables define the nature of the biosludge that enters the system from other processes independent of Unit BX.

The bioslude is loosely defined as fifteen (15) weight percent solids and eighty-five (85) weight percent water. However, this may not always be the case depending on the quality of the biosludge from the wastewater treatment facility. Since, the percent of solids is a key variable to drive other conditions of Unit BX such as lysing agent addition, this variable is measured for the lot of biosludge that is to be processed. The other input variable to the operation of Unit BX, the microbiology of the sludge, influences flowability of the biosludge in the feed hopper and Moyno pump. Since biosludge is an aggregate of dead microorganisms that are employed in the wastewater treatment system, the microbiology play a role in the operation of Unit BX. Since it was discovered late in the project, it was not accounted for. In the middle of July 2004, other processes at

Eastman that pumped the biosludge experienced difficulties as well as Unit BX. It was later determined that the primary cause of the inability to pump biosludge was a result of a filamentous bacteria culture developing in the WWT system. The situation was corrected over a period of a couple of days, but it was clear that the morphology of the bacteria had an impact on the operation of the hydrolysis process.

2.3.2 Process Manipulated/Process Controlled Variables

Process manipulated variables are those that control the operation of Unit BX, sustain operating conditions, and determine the quality of the biosludge product. These variables are essentially the mechanical energy inputs into the system. The main variables are sludge flow rate, lysing agent flow rate, hot oil temperature, top and bottom flange temperature, agitation speed, reactor liquid level, and reactor mixture temperature.

Biosludge flow rate is the primary manipulated variable that dictates all other process manipulated variables. This variable is the rate at which biosludge is loaded into Unit BX; it affects the capacity of the system and other process characteristics such as the flowability of the biosludge. This variable dictates the flow rate of the lysing agent into the system, the lysing agent/sludge mixture flow rate, the amount of energy that can be put into the stream, and the amount of product that can be generated.

The lysing agent flow rate (LA) is the rate at which lysing agent is fed into Unit BX. This variable is a function of the sludge flow rate (WS). Equation 1 illustrates how the mass flow rate is directly proportional to the wet sludge flow rate,

$$F_{LA} \propto F_{WS} \quad \textbf{(1)}$$

where F_{LA} is the mass flow rate of the lysing agent solution, F_{WS} is the mass flow rate of the wet sludge. The relationship is expanded between the two flow rates in equation 2,

$$F_{LA} = K_{LA} F_{WS} = \frac{K_{LA} F_{DS}}{\theta} \quad \textbf{(2)}$$

where F_{DS} is the mass flow rate of the dry sludge (DS), K_{LA} is the proportionality constant between the lysing agent flow rate and the mass flow rate of the biosludge and θ is the percent dry solids in the wet sludge. The proportionality constant is a function of the weight percent of the dried solids in the biosludge, the concentration of the lysing agent in weight percent, the design basis factor, the molar to weight ratio of lysing agent to dried sludge (design basis), the molecular weight of the lysing agent, and the density of the lysing agent. Equation 3 illustrates the relationship of these properties for the proportionality constant,

$$K_{LA} = \frac{\mu_{DBLA} D_B MW_{LA} \theta}{C_{LA}} = \frac{\eta D_B \theta}{C_{LA}} \quad \textbf{(3)}$$

where μ_{DBLA} is the design basis specific to the lysing agent (0.70 lbs of 50% NaOH per lb of dry sludge or 0.35 lbs of 98% H_2SO_4), D_B is the design basis multiple , MW_{LA} is the molecular weight of the lysing agent, θ is the percentage of dried solids in the wet sludge, and C_{LA} is the concentration of the lysing agent solution. η is the combination of the design basis of the lysing agent and the molecular weight of the lysing agent. The K_{LA} factor is the ratio that is entered into the control system that conjoins the lysing agent flow rate with the biosludge flow rate.

Heat is transferred into the reactor by a heat transfer agent. The heat transfer agent is an oil. The term hot oil is used to emphasize that it is the carrier of energy to the reactor. The hot oil system is independent of the operation of Unit BX other than the transfer lines connected to the oil jacket around the reactor. It is a manipulated variable in the operation of Unit BX because it transfers energy through the jacket of the reactor to the contents inside of the reactor. This system controls the final temperature of the reaction mixture (e.g. the temperature the mixture is supposed to reach prior to being quenched). This variable is also coupled with the flow rate of the sludge since the heat transfer from one medium to the other is a function of the flow rates of the two mediums, the difference of temperatures of the inlet and outlet streams of each medium, and the specific heats of the mediums. The rate of heat transfer is a function of the resistances to heat transfer, the surface area, and the logarithmic mean temperature difference of the streams. Since the flow rate of the heat transfer medium is not known, the temperature of the hot oil and the flow rate for the sludge-lysing agent mixture and the liquid level of the liquid in the reactor are matched experimentally such that desired temperature conditions are reached during operation.

Heat loss from the reactor is controlled by a thick layer of insulation around the reactor; however, the top and bottom flanges are not be covered for practical maintenance reasons. Substantial heat loss is possible due to the open surface area to the surroundings and the large temperature difference between of the equipment and the air. To avoid having these exposed surfaces act as heat sinks from the hot oil jacket around the reactor and to concentrate the heat transfer into the mixure inside of the reactor, top and bottom flange heaters were installed to minimize heat loss. These devices add heat to the flanges

to control heat loss. These flange heaters are set to the same temperature as the hot oil.

Agitation of the reaction mixture is important to ensure a uniform product and aids convection with the reactor jacket leading to optimal reactor conditions, which results in a homogeneous bulk reacted mixture. The agitator in the system is a magneto drive agitator, meaning that it is driven by a network of magnets on the agitator shaft and a motor external to the reactor. This is a manipulated variable to the Unit BX. Throughout the study it was kept at 60% operation (approximately 500 rotations per minute) to ideally maintain a consistent mixture of the product.

The reactor liquid level is a controlled variable in the operation of Unit BX. This variable controls the biosludge/lysing agent reactant mixture flow rate into the reactor and the release of the product via the subsequent control valve network. This variable also controls the capacity of the system, the rate of heat transfer due to the contact of the reactor mixture with the sidewalls of the reactor, and the residence time that the reactant mixture is in the system. The liquid level is measured by a gamma ray nuclear emitter and detector on each side of the reactor 180º apart. The liquid level device was calibrated with a graduated volume of liquid such that it will register the volume in the reactor. This variable is significant in that the reactor fills from the top and drains from the bottom. It is crucial to maintain a liquid phase seal to keep a high vapor space pressure in the system so that high temperatures can be reached above the boiling point of water. Additionally, this variable is used to prevent over filling of the system while maintaining optimal heat transfer.

The reactor mixture temperature is the temperature that the reactor mixture is maintained at until the product is discharged from the reactor, quenched in the product

cooler, and released through the control valve assembly. It is desirable that the product temperature reach the design temperature of the experiment.

The valve control assembly is a controlled and manipulated variable used to maintained process conditions. Throughout the duration of the research period, several arrangements were used that would either manipulate the percentage of valve opening through one control valve, through both control valves, or have two valves in sequence where one valve was open while the second valve was closed. Due to the non-Newtonian nature of treated biosludge product, the later method was used. This method is termed blow-case valve control. The control valve process is essentially two valves in sequence with a vertical standpipe that has a vapor space between the valves. The exit valve closes first. The entrance valve then opens to the system, forcing the liquid into the vapor space from the system, compressing the gas in the vapor space from the internal system pressure. The valve at the entrance closes. Then the exit valve opens, allowing the compressed gas in the vapor space to expand, expelling the liquid contents and restoring the blow-case valve assembly to atmospheric pressure. The control element of the setup is a flush cycle, which is the harmonics or the rate at which the valves open and close while maintaining the liquid level in the system. The combination of the timing and the system pressure maintains the liquid level and system pressure.

2.3.3 Post Operation Measured Variables

After the biosludge is reacted with the lysing agent, measurements are made on product sample. These measurements are used to ascertain various qualities of the hydrolyzate such as solids content, viscosity, carbon content, and the oxygen requirement

for biological processes. The measurements (performed on samples that were generated at time intervals), include lysing agent addition relative to the design basis and the reaction temperature, at which the biosludge-lysing agent mixture is maintained. These tests are: the total solids, pH, viscosity, total organic carbon, biological oxygen demand, and titration of the sludge with WWT system influent. Additional tests will be covered in the later chapter for hydrolyzate/water coal slurry adaptation.

The total solid content of the hydrolyzate is the weight percent solids of the biosludge-lysing agent product. This measurement is used in determining a recipe for the treated biosludge/water coal slurry adaptation.

The pH of treated biosludge is an indicator of the basicity/acidity of the product from Unit BX. This value is used as an indication that the lysing agent supply system is working properly.

Viscosity is a metric used to assess flowability of a fluid. One of the primary qualities of the untreated sludge is its very high unmeasurable viscosity and cake-like consistency. The treated sludge has a measurable viscosity profile that is different from that of the untreated state. Topics to address are the kind of non-Newtonian profile the treated material resembles and what is the difference in effect(s) associated with one lysing agent and another lysing agent, if any.

Total Organic Carbon (TOC) and Biological Oxygen Demand (BOD) represent the carbon content of the material and the oxygen requirement for biological treatment. These two variables are to assess the possibility of recycling the treated hydrolyzate to WWT.

Titration of the wastewater influent with the treated biosludge is used to assess

how much treated sludge could be used to raise the pH of an acidic wastewater influent to a neutral pH or conversely lower the pH of the influent and assess how much additional calcium oxide will be needed to neutralize the acidic hydrolyzate. This variable will be described in further detail in chapter 3 for a scaled biosludge treatment unit with recycle to a wastewater treatment facility.

Overall, these variables are used to assess properties of the treated biosludge product from Unit BX and to assess compatibility with other product application feasibility studies. Alteration of these was assessed to determine if there is an operating strategy that leads to an optimal/predictable product.

2.4 EXPERIMENTAL PLAN OF UNIT BX

The experimental plan consisted of start-up of Unit BX, creation of a stable sludge processing system, and performing experiments. Table 2 illustrates the projected timeline for the operational plan. Table 1 translates the operation of Unit BX into 7 separate stages of testing and operation during the summer of 2004 in weeks. Weeks 1 and 2 are to leak test the system and tune the system to operate with water. Weeks 3 through 8 are for introducing biosludge and caustic into the system and performing the appropriate tunings. Week 9 is for optimizing biosludge and caustic for wastewater treatment. Weeks 10 through 13 are for performing catalyst minimization studies with caustic and running the demonstration unit continuously. Week 14 is for sulfuric acid testing. Table 3 outlines the detailed layout of the experimental studies to be run with the system to generate experimental samples.

TABLE 2 Experimental Timeline for the Demonstration Unit

Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
A														
B														
C														
D														
E														

A) Test unit with water feed.
B) Introduce sludge and caustic catalyst and modify process as needed to improve operability.
C) Optimize process at design basis conditions with caustic catalyst
D) Catalyst minimization study for caustic catalyst and demonstration of continuous processing
E) Optimize design basis conditions with sulfuric acid catalyst

TABLE 3 Experimental Plan for Demonstration Unit

Temperature ^{o}C	Catalyst as % Design Basis	Sampling Times in Hours	Lysing Agent(s)
170	200	2, 4, 6, & 8	NaOH
170	100	2, 4, 6, & 8	NaOH
170	75	2, 4, 6, & 8	NaOH
170	50	2, 4, 6, & 8	NaOH
160	200	2, 4, 6, & 8	NaOH, H_2SO_4
160	100	2, 4, 6, & 8	NaOH, H_2SO_4
160	75	2, 4, 6, & 8	NaOH, H_2SO_4
160	50	2, 4, 6, & 8	NaOH, H_2SO_4
150	200	2, 4, 6, & 8	NaOH
150	100	2, 4, 6, & 8	NaOH
150	75	2, 4, 6, & 8	NaOH
150	50	2, 4, 6, & 8	NaOH
140	200	2, 4, 6, & 8	NaOH
140	100	2, 4, 6, & 8	NaOH
140	75	2, 4, 6, & 8	NaOH
140	50	2, 4, 6, & 8	NaOH

2.5 EXPERIMENTAL PROCESS AND DATA

2.5.1 Raw and Treated Biosludge Total Solids Measurement

The process of obtaining total solids of raw or treated biosludge value was performed in three steps: initial weighing of a wet raw or treated biosludge sample, drying of the sample for a period overnight or for 24 hours, and weighing of the dry raw or treated biosludge sample.

The first step was to take three aliquots of untreated biosludge or well-mixed treated hydrolyzate sample samples and place in a tared thermally stable drying container. The mass of the tare and the wet sample was determined on an analytical balance. The aliquots of samples were placed in a drying oven maintained at 104 ^{o}C for a period of 24 hours. The samples were removed from the oven and allowed to cool to room temperature. After cooling, the samples were again weighed on an analytical balance. The difference of the mass after and before drying constituted the total moisture removed from the sample. The ratio of the mass of moisture that was removed and the mass of the wet sample was the weight percent moisture. One hundred percent minus the weight percent of the moisture of the sample is the weight percent of solids in the sample. This test was performed in three times to obtain an average and a standard deviation. The average was the reference of the total solids in all of the treated sludge. The results of these tests were averaged for the experiment.

2.5.2 Flow Rate Conditions for Lysing Agent Addition

After determining the total solids content as discussed in section 2.5.1, the lysing agent to wet sludge ratio was calculated. From this calculation, the flow rate of the lysing agent was determined. The following example shows the detailed steps for calculating the lysing agent to wet sludge ratio that were not illustrated through equation 3 in section 2.3.2

Table 4 outlines all the calculation variables for the lysing agent to wet sludge determination and the appropriate units for the procedure.

Equation 4 is the determination of the dry solids flow rate derived from wet sludge flow rate and the dried solids content of the wet sludge.

$$\frac{A\,g_{WS}}{\min}\left|\frac{B\,g_{DS}}{1\,g_{WS}} = C\,\frac{g_{DS}}{\min}\right. \qquad \textbf{(4)}$$

The product of equation 4 and the design basis of the experiment are used in equation 5 to acquire the flow rate of a pure lysing agent (no water).

$$\frac{C\,g_{DS}}{\min}\left|\frac{D\,g_{LA}}{1\,g_{DS}} = E\,\frac{g_{LA}}{\min}\right. \qquad \textbf{(5)}$$

The product of equation 5 is then used in equation 6 to determine a molar flow rate of the lysing agent to achieve the appropriate rate of addition of the lysing agent to the biosludge flow rate.

$$\frac{E\,g_{LA}}{\min}\left|\frac{1\,mol_{LA}}{F\,g_{LA}} = G\,\frac{g-mol_{LA}}{\min}\right. \qquad \textbf{(6)}$$

The molar flow rate and the molarity of the lysing agent solution are then combined as shown in equation 7 to yield the volumetric flow rate of lysing agent.

TABLE 4 Nomenclature of Generic Variables Used for Lysing Agent to Wet Sludge Determination Example

Generic Variable	Description	Units
A	Mass flow rate of wet sludge	$g \cdot min^{-1}$
B	Percent of dry Solids	unitless
C	Mass flow rate of dry sludge	$g \cdot min^{-1}$
D	Ratio of lysing agent to dry sludge	$g_{LA} \cdot g_{DS}^{-1}$
E	Mass flow rate of 100% lysing agent	$g \cdot min^{-1}$
F	Molecular weight of lysing agent	$g \cdot g\text{-}mol^{-1}$
G	Molar flow rate of 100% lysing agent	$g\text{-}mol \cdot min^{-1}$
H	Molarity of lysing agent solution	$g\text{-}mol \cdot L^{-1}$
I	Volumetric flow rate of lysing agent solution	$L \cdot min^{-1}$
J	Density of lysing agent solution	$g \cdot mL^{-1}$
K	Mass flow rate of lysing agent solution	$g \cdot min^{-1}$
M	Lysing agent solution to wet sludge ratio	$g_{LA} \cdot g_{WS}^{-1}$

The molarity for 50% sodium hydroxide is 18.99 g-mol·L^{-1}.The molarity for 98% sulfuric acid is 18.4g-mol·L^{-1}.

$$\frac{G\ g-mol_{LA}}{\min}\left|\frac{1L_{\ LA\,Solution}}{H\ g-mol_{\ LA}}=I\frac{L_{\ LA\,Solution}}{\min}\right. \qquad \textbf{(7)}$$

The volumetric flow rate is then combined with the density of the lysing agent solution to yield the mass flow rate of the solution. For 50% sodium hydroxide solution at room temperature, the density is 1.52 g·mL^{-1}. For 98% sulfuric acid, the density is 1.84 g·mL^{-1}.

$$\frac{I\ L_{\ LA Solution}}{\min}\left|\frac{J\ g_{\ LA Solution}}{1mL_{\ LA Solution}}\right|\frac{1000\,mL_{LA Solution}}{1L_{\ LA Solution}}=K\frac{g_{\ LA Solution}}{\min} \qquad \textbf{(8)}$$

Lastly, the ratio of the lysing agent mass flow rate and the mass flow rate of the wet biosludge are combined in the manner shown in equation 9 to yield the lysing agent solution flow rate to the wet sludge flow rate ratio.

$$\frac{K\ g_{\ LA Solution}}{\min}\left|\frac{1\min}{A\,g_{WS}}=M\frac{g_{\ LA Solution}}{g_{WS}}\right. \qquad \textbf{(9)}$$

The product of Equation 9 is used to control the lysing agent flow rate relative to the mass flow rate of the wet sludge. Example 1 illustrates the calculation to determine the product as shown in equation 9.

Example 1 Determination of the Lysing Agent to Wet Sludge Ratio for NaOH

Given Variables for NaOH Lysing agent

D = 0.35 g NaOH·g dry sludge^{-1}
F = 40 g·mol^{-1}
H = 18.9999 mol ·L^{-1}
J = 1.52 g ·mL^{-1}

Determined Variables of the Biosludge

A = 500 g·min^{-1}
B = 0.163 g dry solids·g wet sludge^{-1}

What is the mass flow rate of the 50% caustic solution and the caustic to wet biosludge feed ratio?

$$\frac{500\,g_{WS}}{1\,\text{min}}\left|\frac{0.163\,g_{DS}}{1\,g_{WS}}=\frac{81.5\,g_{DS}}{\text{min}}\right.$$

$$\frac{81.5\,g_{DS}}{1\,\text{min}}\left|\frac{0.35\,g_{NaOH}}{1\,g_{DS}}=\frac{28.525\,g_{NaOH}}{\text{min}}\right.$$

$$\frac{28.525\,g_{NaOH}}{1\,\text{min}}\left|\frac{1g-mol_{NaOH}}{40\,g_{NaOH}}=\frac{0.713125\,g-mol_{NaOH}}{\text{min}}\right.$$

$$\frac{0.713125g-mol_{NaOH}}{1\,\text{min}}\left|\frac{1\,L_{50\%\,NaOH}}{18.999\,g-mol_{NaOH}}=\frac{0.0375329\,L_{50\%\,NaOH}}{\text{min}}\right.$$

$$\frac{0.0375329\,L_{50\%\,NaOH}}{\text{min}}\left|\frac{1.52\,g_{50\%\,NaOH}}{1\,mL_{50\%\,NaOH}}\right|\frac{1000\,mL_{50\%\,NaOH}}{1\,L_{50\%\,NaOH}}=\frac{57.05\,g_{50\%\,NaOH}}{\text{min}}$$

$$M=\frac{57.05\,g_{50\%\,NaOH}}{1\,\text{min}}\left|\frac{1\,\text{min}}{500\,g_{DS}}=0.1141\frac{g_{50\%\,NaOH}}{1\,g_{DS}}\right.$$

2.5.3 Measurement of Operation Variables

During the operation of Unit BX, variables outlined in section 2.3.2 are continuously monitored by a computer system called the Pi system. This system takes measurements from field instruments on a fixed time interval and stores the readings for further analysis. The time that the information is stored is approximately 3 years. The data that is stored in the Pi system (often referred to as a historian) is then examined over a selected time interval to obtain pertinent information concerning how the process operated. In the case of Unit BX, the information was accessed at the times the samples were taken. The data was accessed on a period of ten minutes when the samples were

collected. This data was averaged, providing a snapshot of the system each time a sample was taken.

The variables that were monitored during the research period were the reactor temperature, reactor pressure, biosludge flow rate, lysing agent flow rate, hot oil supply temperature, hot oil return temperature, top flange temperature, bottom flange temperature, and reactor level. The residence time is the ratio of the liquid level and the total flow rate of the wet sludge lysing agent mixture. Table 5 illustrates a template of how the data for each sample is summarized in Appendix A. Additional information of the sample include the sample name, the sample date, the design basis for the lysing agent addition, the experimental reaction test temperature, the start time of the data acquisition, the end time of the data acquisition, the time interval (resolution) of the data taken.

TABLE 5 Template of the Data Summary of Each Sample for Appendix A

Sample ID:		Sample Date:		
Sample Start Time:		Sample Finish Time:		
Lysing Agent:		Design Basis:		
Experiment Temp:		Sample Time:		
System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:		±		^{o}C
Reactor Pressure:		±		psig
Flow Rate of Sludge:		±		$gmin^{-1}$
Flow Rate of Caustic:		±		$gmin^{-1}$
Hot Oil Supply Temperature:		±		^{o}C
Hot Oil Return Temperature:		±		^{o}C
Top Flange Temperature:		±		^{o}C
Bottom Flange Temperature:		±		^{o}C
Reactor Level:		±		mL
Residence Time:		±		min
Caustic Sludge Ratio:		±		unitless

2.5.4 Treated Sludge pH Measurement

The pH of a solution is a measure of the hydrogen ion content. It is based on a logarithmic scale between one (1) and fourteen (14) with one being the acidic range of the pH scale and fourteen being the basic range of the scale. For the operation of Unit BX, the pH is an indicator that the lysing agent is being added because the lysing agent is either an acid (H_2SO_4) or a base (NaOH) and that the lysing agent addition system is working. The general procedure for measuring pH was calibrating the pH meter for the range the measurement is to be made in, taking the measurement, followed by cleaning the probe for storage.

2.5.5 Treated Sludge Viscosity Measurements

Viscosity is the ratio of the change of the velocity profile of a liquid through the distance between two parallel surfaces when a shear stress is applied to the liquid. It is used to characterize a fluids resistance to flow. One method to measure viscosity of a fluid is by using a device called a viscometer. One type of viscometer consists of a cylindrical container with a concentric cylindrical shaft (spindle) that is connected to a motor and a torque measurement device. The spindle is rotated at a constant speed (submersed in the fluid) and a torque measurement is made. The torque measurement is then correlated with a viscosity of a Newtonian reference liquid, the type of spindle, and the speed of the motor to yield the viscosity of the fluid that is being measured. The viscosity measurement of the treated sludge was measured with this type of viscometer.

The viscometer used was a Brookfield DV-II+. The types of spindles used to measure the viscosity of the treated hydrolyzate are: LV-1 spindle, LV-2 spindle, LV-3 spindle, LV-4 spindle, and a YULA-15 spindle. The YULA-15 spindle was used with the ultra low viscosity adapter, whereas the other spindles were used with a 600 mL Beaker. The spindle was selected based on whether the viscosity of the liquid was within the range for the spindle and the speeds used. The general procedure was to attempt to measure eight viscosity measurements at different speeds. When the viscosity became steady, the measurement was taken. The percent speed, percent torque, and viscosity were recorded.

2.5.6 Treated Sludge Total Organic Carbon Measurements (TOC)

Total Organic Carbon (TOC) is a test to measure organic carbon in a sample. Carbon compounds can occur in two forms, either inorganic carbon in the form of carbonate salts or organic carbon in organic compounds. The organic compounds can in turn be used by microorganisms as an energy source and/or a source of carbon to construct macromolecules necessary for a microorganism's cellular functions. Equation 10 illustrates the relationship of the total inorganic carbon (TIC), Total Organic Carbon (TOC), and Total Carbon (TC).

$$TIC + TOC = TC \tag{10}$$

Upon heating the sample to a high temperature in the presence of oxygen, all organic carbon compounds will convert to carbon dioxide with oxygen or decomposition of an

inorganic carbonate salt will liberate carbon dioxide from the heat. This situation presents a problem in that liberation of carbon dioxide from the inorganic carbonate salts will cause an inaccurate calculation of the organic carbon content of a sample. However, lowering the pH of a sample with a concentrated acid to prior to combustion releases the carbon dioxide prior to testing of the sample for the organic carbon content eliminates this problem. Typically, the samples are titrated with a solution of phosphoric acid to liberate the carbon dioxide from the carbonate salts. Then the sample is heated to a high temperature to liberate the carbon dioxide from the organic molecules via oxidation, and then the carbon dioxide is measured to determine the carbon content of the sample.

This is the general method used to measure the TOC. The tests of selected samples were tested by Eastman laboratories to determine the carbon content of the treated sludge. The use of the information from these tests are further examined in Chapter 3.

2.5.7 Treated Sludge Biological Oxygen Demand (BOD)

Biological Oxygen Demand (BOD) is a measurement to determine the oxygen required for microorganisms to consume organic compounds in a sample. For aerobic biological processes, oxygen consumption is an important factor to consider. The limitation of aeration is the amount of oxygen that can be dissolved into water. The limit is approximately 7ppm. So, the size of a biological treatment system is ultimately constrained by the amount of oxygen it can deliver via mechanical aeration.

The goal of performing a BOD test is to determine the theoretical oxygen demand for microorganisms to consume all of the organic material. The typical procedure to test

BOD is to take an analytically diluted sample mixed with a microorganism "seed" standard and well aerated water and combine. This combination is allowed to sit for a prescribed period of time while the microorganisms consume the organic compound(s). At the initiation of the test, the dissolved oxygen is measured. Maximum consumption of oxygen for these tests is approximately 3 ppm so as to prevent anoxic conditions in the test for the microorganisms to utilize nitrate or nitrite salts as the terminal electron acceptor for the biological reaction of the organic compound(s). So, the minimum of the dissolved oxygen is approximately 4 ppm. The mixture of the sample, microorganisms, and well aerated water is allowed to sit for a prescribed period of time before the dissolved oxygen is measured. Once the difference of the beginning and ending dissolved oxygen concentrations is known, the value is scaled by the dilution factor.

The dilution is used to constrain the consumption of oxygen within the dissolved oxygen range of 7 ppm and 4 ppm. Typically, multiples of these tests are performed at the same time to ascertain the appropriate dilution factor. Additionally, the period of time that the samples are allowed to sit is important. For example, if the tests for a sample are allowed to sit for a period of 5 days with all other factors held constant and another set of samples were allowed to sit for six days, the comparisons of the test results would most likely be invalid. Therefore, all tests must be performed over a similar duration. The presence of a subscript is typically the number of days that the test allowed to run. For example Biological Oxygen Demand 5 days will be noted as (BOD_5).

For the samples that were examined, BOD_5 tests were to be used. There is an additional notation to be considered. This term is BOD_U, which is the called ultimate BOD. This value is determined by alloying the solution with the sample and the

microorganisms to sit for a very long period of time for the microorganisms to consume all of the organic material. Experience from Eastman personnel and Metcalf and Eddy[2,17] suggest that the dissolved oxygen consumed during the 5 day test is approximately 70% of the total BOD or BOD_U. The tests for this examination were performed at Eastman Laboratory.

2.5.8 Treated Sludge and WWT Influent Titration

If the treated hydrolyzate were recycled to WWT (discussed in chapter 3) the question becomes how much calcium hydroxide could be offset or required with the type of lysing agent treatment used. The treated hydrolyzate has either a high or low pH depending on whether the lysing agent is a base or an acid. The goal of this test is to assess the amount of sludge that could mix with Eastman WWT influent to raise the pH to neutral (offsetting the use of calcium hydroxide) or lower the pH of the recycle stream to neutral (requiring additional calcium hydroxide with the current demand to raise the pH of the influent stream).

The general procedure for these tests is as follows. A measured sample of WWT influent treated sludge was loaded into a vessel large enough to contain the sample and the influent titrant (for basic treated sludge). A calibrated pH probe/meter was then inserted into the WWT influent as it was mixed by a stirring apparatus. Once an initial pH reading was made, the titrant (treated hydrolyzate) is added until the mixture reaches a pH of 7 and the base pH of the WWT influent is neutralized. The same test was performed, except a 2% calcium hydroxide solution was used in place of the treated

sludge. The ratio of the titrants was then used to determine the amount of calcium hydroxide that could be offset by the addition of the base treated hydrolyzate.

Conversely, for the acidic treated biosludge, the titrant is calcium hydroxide solution of a known concentration. This was added to the acidic treated hydrolyzate until the pH reacheed neutral. The volumetric amount of the calcium hydroxide solution used is then used to determine the additional stoichiometric requirement of calcium hydroxide in addition to the calcium hydroxide used to raise the the pH of the WWT influent.

2.6 RESULTS AND DISCUSSION OF UNIT BX

2.6.1 Summary of Results

Operation of Unit BX at Eastman, Kingsport, TN between May 2004 and August 2004 was a success, but provided many challenges. The successful part of this project resulted in large volume samples were acquired for further testing, continuous operation was acheived for a duration of 62 hours, and key sources of operational upsets were identified. In contrast, optimal operating conditions were not identified, because identification of the key sources of process upsets occurred late in the project, which allowed only enough time to run the remaining experiments. A total of 18 experiments were conducted, 16 for sodium hydroxide lysing agent and 2 for sulfuric acid. A total of 44 two (2) quart samples of sodium hydroxide treated biosludge and 5 two (2) quart samples of sulfuric treated biosludge were created for testing at the University of Tennessee. Drawbacks to operation included finding that the process was labor-intensive lead to premature experiment terminations. Unpredictable process upsets lead to erratic/undesired process conditions sometimes during experimental processing. The

following sections will be: the lysis of the biosludge from the operation of Unit BX, a discussion of the phenomena and processing challenges encountered leading to system upset, strategies used to mitigate process upsets, and a summary of the samples collected with the averaged process conditions of key process manipulated/controlled variables and post operation measurements of selected samples.

2.6.2 Lysis of Biosludge

The hypothesis used in this work is that the lysing agent will rupture the cell wall of the microorganism, releasing the organic macromolecules, cellular structures, and the bound water from the microorganisms that compose the biosludge, creating a nutrient/energy-rich product with a water-like consistency. Figure 18 illustrate the lysing phenomena of the treated biomass from Unit BX processed samples compared to the untreated biomass feedstock. This figure shows the unlysed biosludge on the left-hand side and the treated biosludge on the right-hand side. As seen the untreated biosludge on the left-hand side has a bulk conglomerate dark opaque appearance because of the clusters of agglomerated microorganisms and inert material. In the adjacent photographs on the right showing the treated biosludge, the opaque clusters are significantly reduced in size with the appearance of inert material also shown as dark matter. The translucent articles are the cell walls of the microorganisms and the transparent voids are the water and nutrients contents of the biomass. These figures illustrate the nature of lysis that is used for study in other applications and to support the hypothesis that this research is focused on.

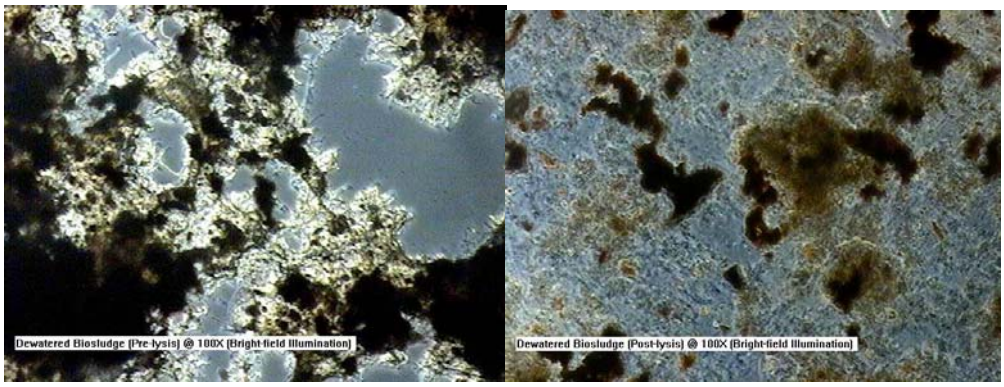
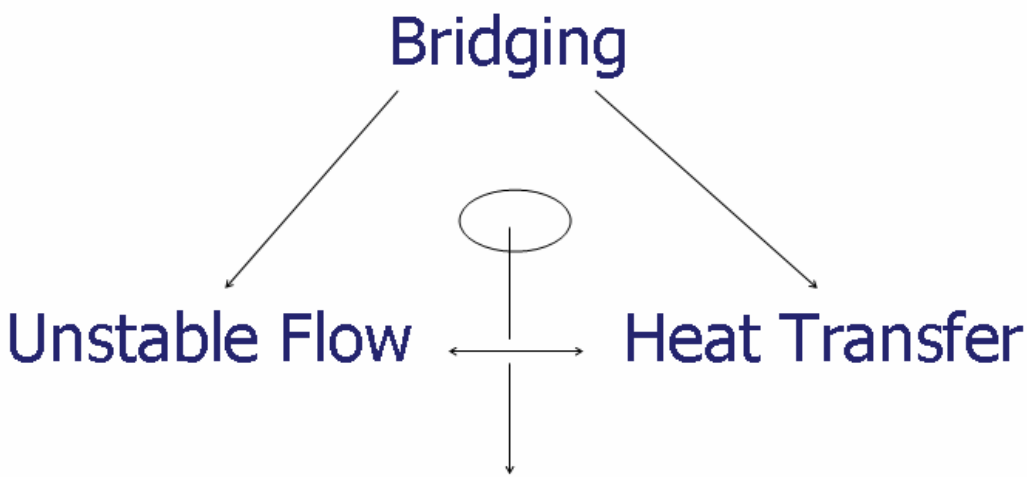


FIGURE 18 Comparison of Pre and Post-Lysis at 100 X BFI

2.6.3 Challenges Encountered in Operation of Unit BX and Strategies to Overcome Processing Challenges

In the testing of both lysing agents, operational difficulties were encountered. These difficulties were a result of instability of the operation of Unit BX. The result of these instabilities was either over-pressurization or under-pressurization of the system. Unit BX's operational stability is dependant on three stable operating conditions: dewatered sludge feed, treated sludge flow control, and biosludge/caustic heating. Unstable operation of Unit BX could result from the following: bridging of the dewatered sludge feed, inadequate product flow control, or adverse heat transfer conditions. If any of the cited conditions occur, then Unit BX will over or under pressurize resulting in subsequent mechanical failure and experiment termination. Figure 19 illustrates the relationships of adverse conditions that result in unstable operation. As shown in the diagram, should any of the three instabilities occur, other operating processes of Unit BX would become unstable, in many cases resulting in shutdown or worse emergency



Causes of Rupture Disc Blow out: Liquid Level Control, Pressure Control, Flow Control

FIGURE 19 Primary Disturbances that Result in Experiment Termination.

shutdown. To illustrate the sensitivity of Unit BX to unstable operation, three scenarios will be described. The three scenarios are bridging of the raw sludge in the feed hopper, unstable flow control of the treated sludge, and inadequate heat transfer. Strategies to mitigate the dynamics from these potential scenarios will also be described.

2.6.3.1 Case 1: Raw Sludge Bridging in the Feed Hopper

Bridging is a term used to describe the collection of material in such a manner to form a "bridge" of loose material across a span, such that cavities of air develop beneath the bridge material. These pockets create a problem.

Instead of the raw untreated sludge being loaded into Unit BX with a constant mass flow rate, some air is fed into the system instead. The raw untreated sludge is assumed to be an incompressible fluid, but this is not the case for air. When the air is

loaded into Unit BX under pressure one of two upsets could occur. The system could "hiccup" causing unstable flow control of the treated product or the material flow could stop completely allowing for build up of heat in the fluid causing other system upsets.

In the case of an intermittent "hiccup" of the system, the flow would lead to one of two scenarios: either complete draining of the reactor or over pressurizing the system. If the reactor were to completely drain, then the feed entering the reactor could potentially flash boil because the liquid seal is no longer a seal caking solids at the exit and plugging the reactor. The result would be over pressurization of the system and bursting of the rupture disc. If the system over-pressurizes a rupture disc could burst, resulting in termination of the experiment and the need for subsequent equipment repairs.

In the case of the flow of raw untreated biosludge stopping, the release of the treated biosludge stops due to level control management and allows excessive heating of the treated product in the CSTR. The untreated sludge in the lines begins to heat up to the point that the vapor pressure of the water will exceed the pressure rating of the system. The end-result is a ruptured rupture disc, termination of the experiment, and the need for subsequent equipment repairs.

The strategy used to eliminate this type of system upset was the use of a paddle like tool. The paddle-like tool was hand operated to push around the raw sludge to evacuate any air cavities and break up bridged sludge. The end-result being continued system operation for 62 hours instead of a typical 4-6 hour run operation.

2.6.3.2 Case 2: Unstable Treated Sludge Flow Control

Unstable flow control is encountered when either the sludge drains out of the reactor faster than the liquid is added to the reactor or the treated sludge does not drain from the reactor at all. If the reactor is completely evacuated, the flashing of the feed entering the reactor creates a system "hiccup," which potentially stops the flow of biosludge and leading to over-heating of product and over-pressurizing of the system, bursting a rupture disc and resulting in early termination of the experiment. If the sludge does not drain at all, the system becomes over-pressurized with static liquid pressure delivered by the Moyno pump, causing any of the systems rupture discs to burst. In turn, the experiment is terminated and subsequent repairs are required prior to restarting the system.

Factors that can create this scenario are: foreign material such as screws, agitator paddles, and solid buildup in the system blocking normal system functions; cavities that form in the feed hopper due to bridging of the feed sludge; or the shear thinning properties of the treated sludge, which will be discussed later. Practices that were applied to minimize system upsets of this kind included careful monitoring of the raw sludge for foreign objects, ensuring that system internal equipment was secure and functioning properly, frequent agitation of feed sludge to evacuate any air pockets was employed, and a using the blow-case valve control strategy to control fluid flow.

2.6.3.3 Case 3: Unsteady Heat Transfer

If heating in the system is either inadequate or too much, then the hydrolyzate mixture temperature would either drop below experimental operating temperatures or the

hydrolyzate mixture temperature would rise above the maximum system operating temperature.

If the hydrolyzate mixture temperature drops too much, the viscosity of the treated sludge becomes high. An impact of increased fluid viscosity is that flow control would become unstable, stop the flow of the sludge, and cause flooding of the system. This would cause the rupture disk to burst, causing early experiment termination and the need for subsequent system equipment repairs.

If the hydrolyzate mixture temperature becomes too high, the pressure would rise above the system maximum operating pressure. In turn, the rupture disk above the CSTR would burst causing early experiment termination and the need for subsequent equipment repairs.

2.6.3.4 Strategies for Improved Process Management of Unit BX

Throughout all experiments, three key operational strategies were used to minimize experiment disruption. These strategies are feed sludge bridging break up, sludge lysis preheating, and blow-case valve flow control.

Feed sludge break-up consists of using a paddle like tool to break up air pockets and free bridged material from the sides of the feed hopper. Systematic application of this simple device resulted in a substantial reduction of system upsets. The period of application was typically between 15 and 30 minute intervals during the experimental run or when an upset was in flow rate of the untreated sludge was beginning.

Sludge lysis preheating involves the addition of steam tracing to the pipes prior to the CSTR inlet. Additional equipment used included several temperature indicators at

different points of the system as well as insulation of the traced pipes to keep heat from escaping. This strategy helped in maintaining hydrolyzate mixture temperature in the event of the CSTR completely drained or the loss of control of the hydrolyzate mixture residence time.

Throughout the research period six different control strategies of the liquid flow rate were used, but only one was successful. The six strategies tested were

1. Level control using CV2 only in the horizontal orientation
2. Level control using CV1 to control pressure and CV2 for controlling level
3. Level control with CV1 in the vertical orientation after the heat exchanger and CV2 in the horizontal orientation pressure with a flush cycle
4. Blow case with CV1 in the vertical orientation and CV2 in the horizontal orientation
5. Blow case with CV1 and CV2 at a 45º incline
6. Blow case with CV1 and CV2 on a 45º incline with the addition of a large stand-pipe

(Diagrams of these strategies are shown in Appendix B)

Strategy 6, the blow case with CV1 and CV2 on a 45º incline with a large stand-pipe, was the solution to controlling the sheer thinning properties of the sludge. Originally, the system was designed and tested with water, which is a Newtonian fluid. Since sludge is primarily water, it was assumed that the treated product would exhibit similar characteristics and constant viscosity with shear rate. This turned out not to be the case for treated biosludge. The treated biosludge displays sheer thinning properties, meaning that when a shear stress is applied to the fluid the viscosity drops. The end-result of this behavior was that the control system that was in place would have to remove aliquots of sludge periodically rather than in a continuous manner. This is the primary basis of the the blow case system. While CV2 is closed, CV1 opens to the system side allowing liquid to enter into the vertical stand pipe, compressing the vapor space in the standpipe

to a smaller volume and the remaining vapor space filled with treated product. CV2 closes from the system and CV1 opens allowing the vapor in the vapor space to expand, pushing the trapped liquid out of the vertical stand-pipe. CV2 closes and the process is repeated.

Overall, the strategies described led to successful continuous operation of Unit BX for a period of for 62 hours. The strategies did not necessarily erradicate processing difficulties, but minimized subsequent equipment damage and complete process shutdowns allowing the study to be completed.

2.6.4 Experimental Data

The operation of Unit BX led to a collection of samples in large volumes for further evaluations that could not be achieved with small sample sizes. This section will focus on details of the samples taken during experiments performed using Unit BX showing all the samples that were taken, the identification of the samples, the duration of the experiments, the average conditions of Unit BX while the samples were taken and a subset of the gravitiometric analysis of the samples for both sodium hydroxide and sulfuric lysing agents.

Table 6 shows all the samples taken per unit time of the experiment, the design temperature, and the design basis employed for sodium hydroxide. As seen in Table 6, the temperatures used were 170 ^{o}C, 160 ^{o}C, 150 ^{o}C, and 140 ^{o}C. The design bases used for each design temperature were 200%, 100%, 75%, and 50%. The planned test duration for each experiment was approximately 8 hours, during which samples were taken at approximately 2-hour intervals. In some circumstances, experiments were

TABLE 6 Sample IDs for Each Catalyst Minimization Experiment Samples for Sodium Hydroxide Tests.

Temperature	Design Basis	Time (hours)			
		2	4	6	8
170	2	072104-JH-01	072104-JH-02	072104-JH-03	072104-JH-04
	1	072204-NB-01	072204-NB-02	072204-NB-03	072204-JH-04
	0.75	072304-JH-01	072304-JH-02	Operational Difficulties	
	0.5	070804-NB-01	070804-NB-02	Operational Difficulties	
160	2	080604-MV-08	080604-JH-09	080604-JH-10	080704-JH-01
	1	080604-NB-05	080604-NB-06	080604-NB-07	Operational Difficulties
	0.75	080604-JH-02	080604-JH-03	080604-JH-04	Operational Difficulties
	0.5	080504-MV-06	080504-JH-07	080504-JH-08	080604-JH-01
150	2	072604-NB-01	072604-NB-02	Operational Difficulties	
	1	072704-NB-01	072704-JH-02	Operational Difficulties	
	0.75	072804-NB-01	Operational Difficulties`		
	0.5	080504-NB-04	080504-NB-05	Operational Difficulties	
140	2	080304-JH-01	080304-JH-02	080304-JH-03	Operational Difficulties
	1	080404-NB-01	080404-NB-02	080404-NB-03	Operational Difficulties
	0.75	080404-JH-04	080404-JH-05	Operational Difficulties	
	0.5	080504-JH-01	080504-JH-02	080504-JH-03	Operational Difficulties

terminated due to operational difficulties, so some of the samples could not be taken at desired time intervals. Table 7 shows the dates and times that the corresponding samples from Table 6 were taken for the sodium hydroxide experimental design. Forty-four sodium hydroxide treated sludge samples were taken.

Table 8 shows all the samples taken per unit time of the experiment, the design temperature, and the design basis employed for sulfuric acid. As seen in Table 8, the temperatures used was 160 ^{o}C. The design bases used for each design temperature were 100% and 50%. The planned test duration for each experiment was approximately 8 hours, during which samples were taken at approximately 2-hour intervals. In some circumstances experiments had to be terminated due to operational difficulties, so some of the samples could not be taken at desired time intervals. Table 9 shows the dates and

TABLE 7 Dates and Times that Correspond to Samples from Table 5 that were taken for Sodium Hydroxide Tests.

Temperature	Design Basis	Time (hours)			
		2	4	6	8
170°C	2	7/21/2004 15:40	7/21/2004 17:40	7/21/2004 19:40	7/21/2004 21:40
	1	7/22/2004 15:00	7/22/2004 17:00	7/22/2004 19:00	7/22/2004 21:00
	0.75	7/23/2004 1:19	7/23/2004 3:40	Operational Difficulties	
	0.5	7/8/2004 15:40	7/8/2004 16:40	Operational Difficulties	
160 °C	2	8/6/2004 18:30	8/6/2004 20:30	8/6/2004 22:30	8/7/2004 0:30
	1	8/6/2004 10:50	8/6/2004 14:15	8/6/2004 16:00	Operational Difficulties
	0.75	8/6/2004 4:45	8/6/2004 6:45	8/6/2004 8:50	Operational Difficulties
	0.5	8/5/2004 18:30	8/5/2004 20:30	8/5/2004 10:20	8/6/2004 0:30
150 °C	2	7/26/2004 17:15	7/26/2004 19:10	Operational Difficulties	
	1	7/27/2004 15:45	7/27/2004 17:45	Operational Difficulties	
	0.75	7/28/2004 14:26	Operational Difficulties		
	0.5	8/5/2004 12:15	8/5/2004 14:15	Operational Difficulties	
140 °C	2	8/3/2004 15:05	8/3/2004 17:08	8/3/2004 19:03	Operational Difficulties
	1	8/4/2004 12:15	8/4/2004 14:30	8/4/2004 15:03	Operational Difficulties
	0.75	8/4/2004 20:20	8/4/2004 22:30	Operational Difficulties	
	0.5	8/5/2004 4:00	8/5/2004 6:00	8/5/2004 8:00	Operational Difficulties

TABLE 8 Sample IDs for Each Lysing Agent Minimization Experiment Sample for Sulfuric Acid Tests.

Temperature	Design Basis	Time (hours)			
		2	4	6	8
160°C	1	081104-JH-01	081104-JH-02	081104-JH-03	Operational Difficulties
	0.5	081204-NB-01	081204-NB-02	Operational Difficulties	

TABLE 9 Dates and Times that Corresponding Samples from Table 7 were taken for Sulfuric Acid Tests.

Temperature	Design Basis	Time (hours)			
		2	4	6	8
160°C	1	8/11/2004 16:20	8/11/2004 18:25	8/11/2004 20:45	Operational Difficulties
	0.5	8/12/2004 0:00	8/12/2004 2:00	Operational Difficulties	

times that the corresponding samples from Table 8 were taken for the sulfuric acid catalyst minimization. A total of five sulfuric acid treated sludge samples were taken

As seen in all four tables, the 8-hour experiments could not be completed for all cases. The reasons are a result of the process upsets mentioned earlier. However, the main goal of this project was to first, create an operational system that could run in continuous mode, and secondly generate industrial grade samples for further studies. The following discussion will cover the average system operating conditions when the samples were collected. The average characteristics that will be presented are: the average reactor mixture temperature; average biosludge and lysing agent flow rates; average reactor liquid level; and average total solids of the treated sludge.

Figure 20 illustrates the average reactor mixture temperature with respect to the experiment performed. As can be seen in the graph, the average reactor temperature of each experiment with exception of the lower design basis' experiments at 140º C is within ±5 º C. The anomalous temperature was most likely due to the low concentration of lysing agent creating a more viscous product that did not transfer heat into the system well, elevated utility temperatures for the experiment temperature, and/or high liquid volume in the reactor, which created a maximized liquid contact with the walls of reactor allowing maximum heat transfer into the liquid.

The error bars indicated the standard deviation between samples of samples collected in the experiment. The standard deviation is an indication of the stability of the system at the time the samples were taken. With regard to the average reactor temperature, the experiments that have the largest standard deviation are the 140ºC at

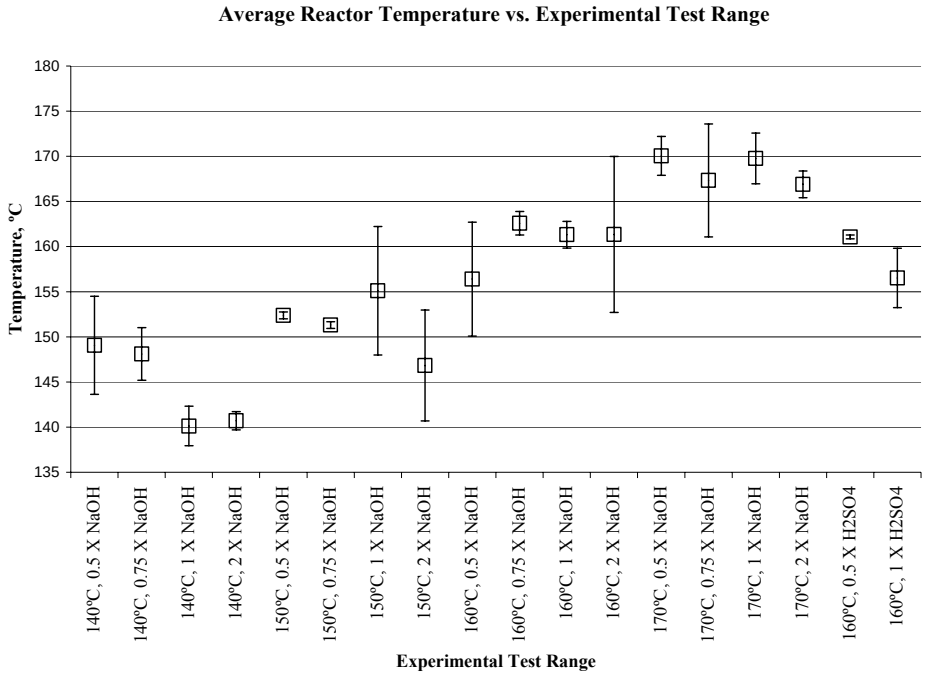


FIGURE 20 Average Reactor Temperature with Respect to Experiment Performed

50%, 150 at 100% and 200%, 160ºC at 50% and 200%, and 170ºC at 75%. Reasons for these deviations are not clear.

Figure 21 illustrates the flow rates of the biosludge and lysing agent entering Unit BX. The average flow rates for the sodium hydroxide lysing studies are in a range of 500 $gmin^{-1} \pm 100\ gmin^{-1}$. For the sulfuric acid experiments the sludge flow rate is in the range of $350\ gmin^{-1} \pm 75\ gmin^{-1}$. The lysing agent flow rates range from 100-125 $gmin^{-1}$ for 200% design basis and 10-25 $gmin^{-1}$ for 50 % design basis for the sodium hydroxide. For the sulfuric acid experiments, the lysing agents flow rates were much lower. This is because the sludge flow rates were lower and the concentration of the lysing agent injected into the stream was 98% concentration of the lysing agent as opposed to 50% sodium hydroxide. As seen, the standard deviation for some experiments were small

with exception to 75% and 100% design basis at 140ºC, 100% and 200% design basis at 150ºC, and 50%, 75%, 100%, 200% design basis at 160ºC. This was primarily due to the variable/unstable flow rates from those measured in the biosludge feed during the sampling of the product. For the deviations for the lysing agent flow rates are small compared to the scale of the graph, less than 10 $gmin^{-1}$.

Figure 22 illustrates the average liquid volume of Unit BX with respect to the experiment run. As seen in the graph the average liquid volume ranges from 4000 mL to 6000 mL. Deviations greater than 400mL are indications that the system was very unstable, that flow settings were changed during operation of the system, or that the release of product was accelerated by erratic control valve operations. Sample sets for which the deviations suggest this are 50% design basis at 140ºC, 100% at 150ºC, 200% at 160ºC, and 100% at 170ºC.

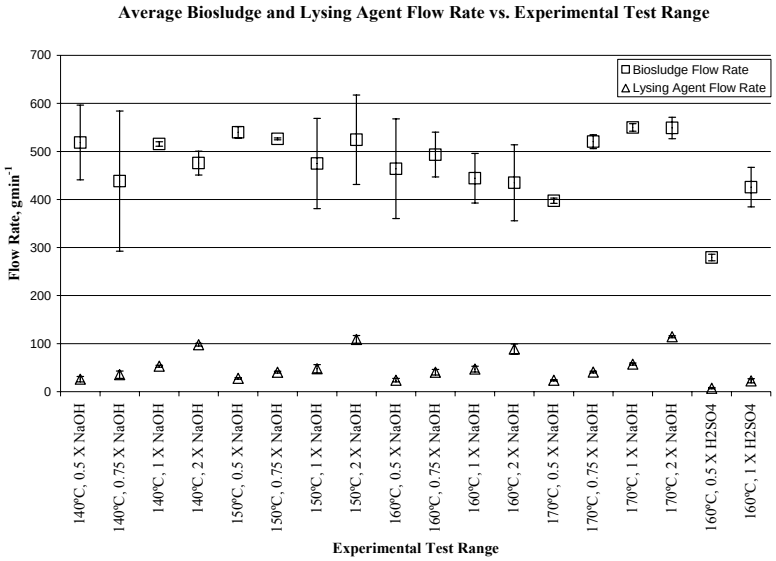


FIGURE 21 Average Biosludge and Lysing Agent Flow Rates with Respect to Experiment Performed

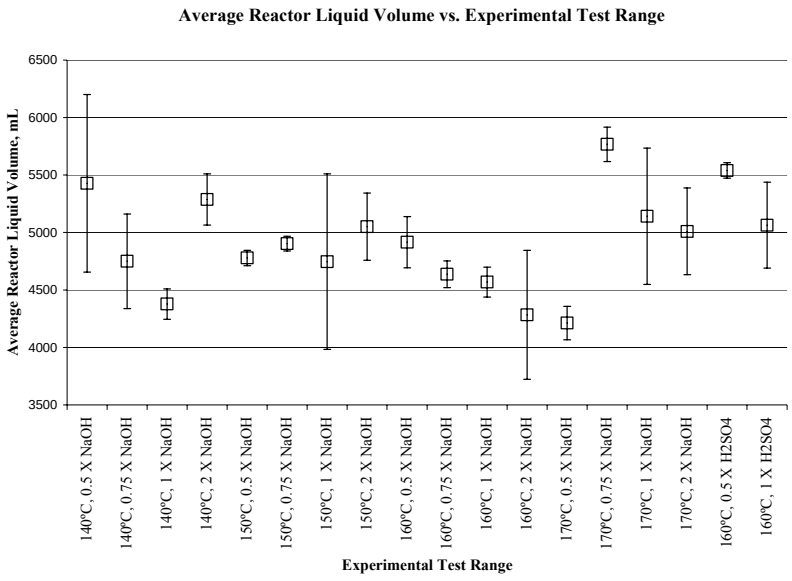


FIGURE 22 Average Reactor Liquid Volume with Respect to Experiment Performed

Figure 23 illustrates the average residence time of the biosludge and lysing agent in the reactor. The residence time is the ratio of the reactor liquid volume and the liquid flow rate. The average residence time of Unit BX for all experiments is approximately 9.9 minutes. For the sodium hydroxide experiments, the average residence time is approximately 9.2 minutes. For the sulfuric acid, the average residence time is approximately 15.4 minutes. Deviations in the residence time suggest system upsets, changes in inlet flow rates from one sample set to another sample set in an experiment, or change in discharge rate from the reactor. Samples that have deviations greater than 1 minute are 50% and 75% design basis at 140ºC, 100% and 200% at 150ºC, 50%, 100%, 200% at 160ºC, and 100% at 160ºC for sulfuric acid. The extremely high residence time for sulfuric acid at 19 minutes is due to low flow rate and high liquid volume in the reactor.

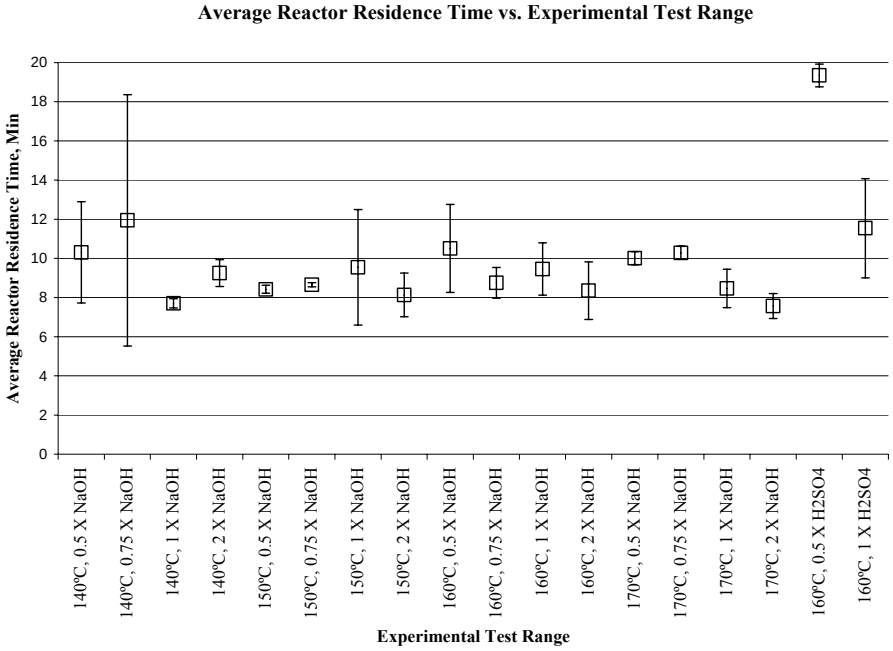


FIGURE 23 Average Reactor Residence Time with Respect to Experiment Performed

The cause of the high residence time for the sulfuric acid tests is the elevated pressure of the system during operation.

Figure 24 illustrates the average reactor pressure at the time of sampling with respect to experiment performed. From saturated steam tables the gauge pressure for each of the experimental temperatures are 37.75 psig, 54.38 psig, 74.99 psig, and 100.22 psig for 140ºC, 150ºC, 160ºC, 170ºC respectively. As seen in Figure 26, the pressures are significantly higher than the steam table pressures with the exception of 200% at 150ºC. The reason the pressures are higher than the steam table pressures for most of the experiments is most likely air being fed into the system via the biosludge, being dissolved into the water into the cold section, being released in the hot sections of the system, and becoming trapped in the vapor space of the reactor were it could not escape. For the

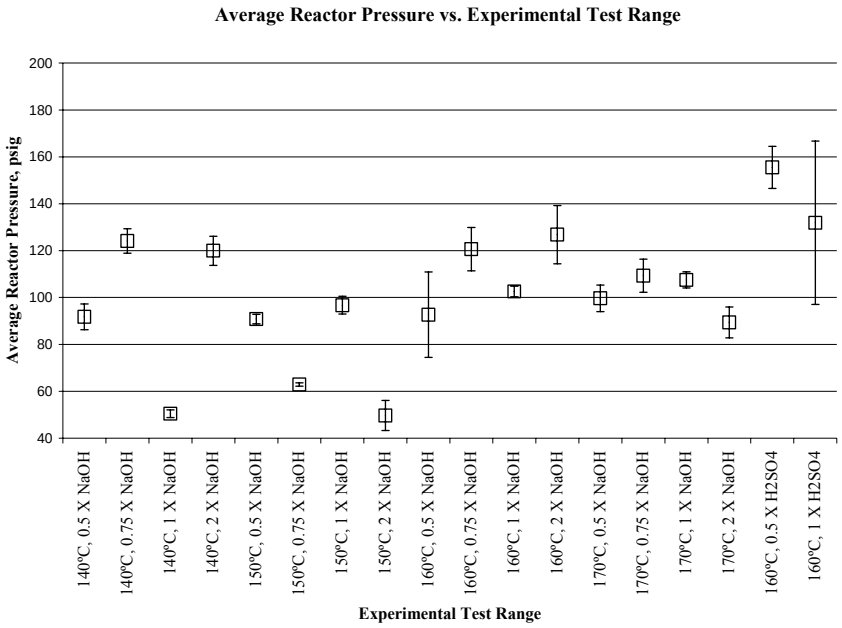


FIGURE 24 Average Reactor Pressure with Respect to Experiment Performed

sulfuric acid experiments, the extremely high pressure greater than 130 psig is most likely a result of a neutralization reaction with carbonate salts present in the biosludge in the form of calcium carbonate. Since the system was not designed to accommodate the accumulation or generation of gases in the system, the system was subject to instability when such took place.

Figure 25 illustrates the total dried solids with respect to the experiment performed. As can be seen the average total solids averaged 15 percent and range from 7 percent to 25 percent. The higher range of the average is most likely a result of high lysing agent concentrations. The lower range is most likely caused by a sample that had been contaminated in the sampling process or, by solids which were allowed to settle, skewing the results of the test. The relationship would be expected to be more like shown

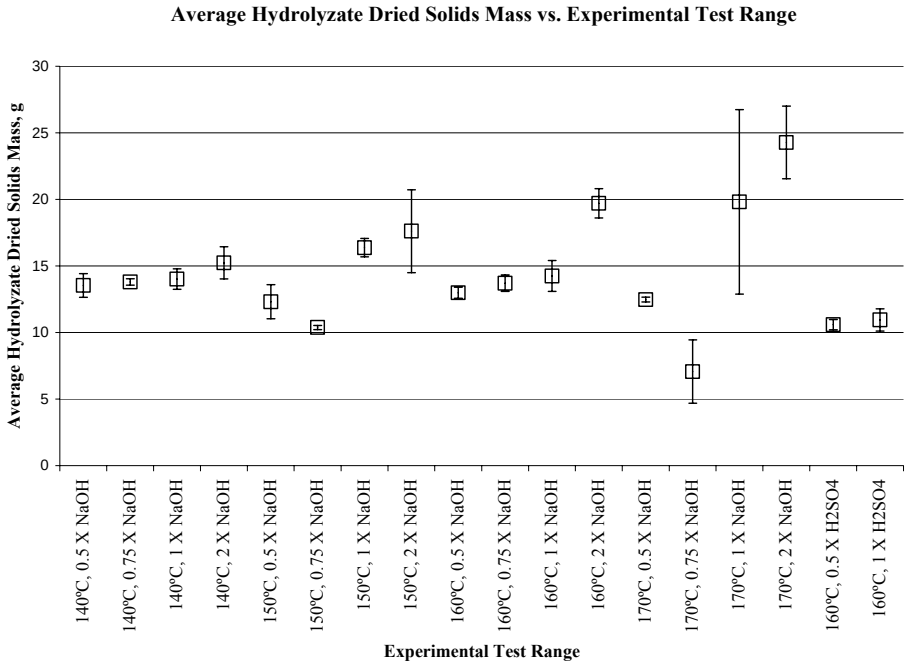


FIGURE 25 Average Hydrolyzate Dried Solids with Respect to Experiment Performed

at 140ºC and 160ºC, where all of the samples were created in a one week period. Other possible causes for deviation from some the expected typical values could be the variations of untreated biosludge solids introduced into the system, where the 50% design basis at 170ºC was created at the beginning of the month of July, and the fact that the 200% design basis at 170ºC was created 2-3 weeks later. It is to be expected that the mass of the higher design basis would be greater than the subsequent samples since the caustic adds to the total solids, therefore leading to the higher mass. The standard deviation of the samples between times and individual tests was approximately 1%. The four cases in which this was not so are 200% design basis150ºC, 75%, 100%, and 200% at 170ºC. These are most likely a result of erratic process operation and stability.

Figure 26 illustrates the viscosities of selected samples with respect to shear rate. The profile that is seen Figure 26 is a typical indicator of a profile of a shear thinning liquid. The hydrolyzate of all samples vary similarly with the magnitudes of viscosity. The viscosities of the sulfuric acid samples were not measured due to the extreme corrosive properties of the treated sludge with the metal parts of the viscometer, affecting the surface finish of the parts. The samples shown above are the samples with the lowest measure of viscosities. The relationship between the incoming raw sludge, the process conditions, and the nature of the treated product is unclear. Overall, the treated biosludge, despite its non-Newtonian characteristics at a low shear rate, can resemble water-like flow properties allowing for simple transport mechanisms compared to the untreated solid biosludge.

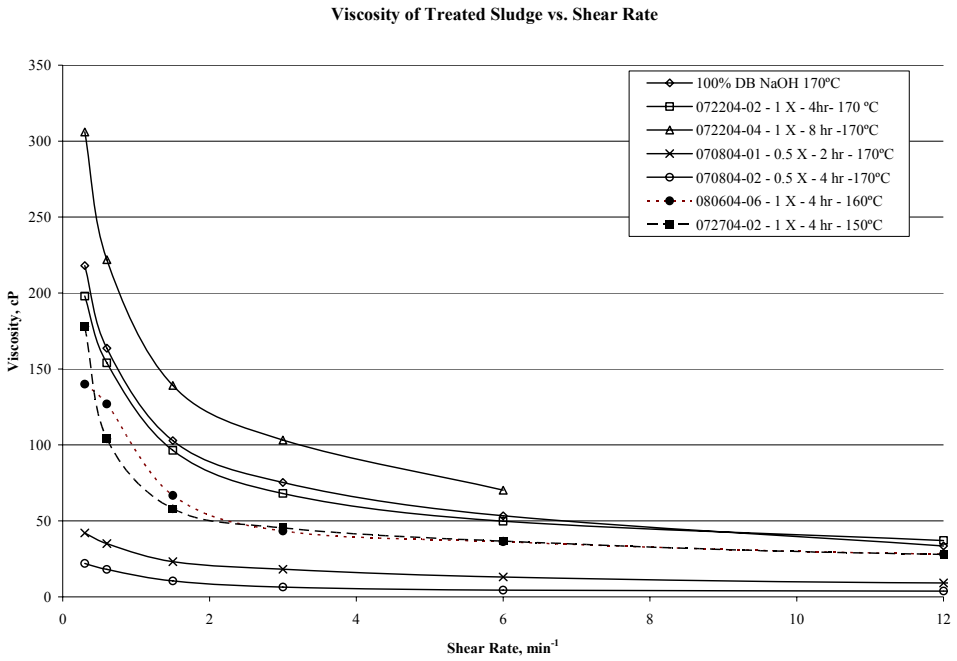


FIGURE 26 Viscosities of Selected Sodium Hydroxide Hydrolysate Samples with Respect to Shear Rate

The BOD_5 and TOC are indicators of the ability to biologically oxidize the organic compounds present in the material or determine the bioavailability of the carbon in the material.

As a consequence of this study, these factors are necessary to model the impacts of recycling treated hydrolyzate to WWT. Table 10 is a tabular representation of the BOD_5 and TOC measurements of a selected set of samples. The design basis that were tested are 50%, 100%, 200% at 160ºC for sodium hydroxide and 50% and 100% at 160ºC for sulfuric acid. As seen the range for the sodium hydroxide samples BOD_5 is from 37,000 to 26,000 mg/L and TOC from 42,500 to 26,800 mg/L. The range for the sulfuric acid samples for the BOD_5 is from 28,000 to 24,000 and TOC from 32,300 to 24,600. The data suggests that the higher the concentration of the design basis resulted in a higher BOD and TOC showing that the lysis and treatment of the sludge particles make the organic compounds more bioavailable.

Table 11 shows the titration of the basic hydrolyzate with WWT influent and the amount of calcium oxide necessary for neutralization of acidity of the acidic hydrolyzate to a neutral pH. Overall, the amount of hydrolyzate required to raise the pH of the 250 mL of WWT influent are 0.30 mL for 200% design basis, 0.517 mL for 100% design basis, and 1.60 mL for 50% design basis at 160ºC. The neutralization of the WWT influent with 1% $Ca(OH)_2$ solution was 2.2mL. The offset from stoichiometric calculations for the offset of a 20% lime solution with a WWT sludge production of 45 dry tons 16 weight percent raw biosludge would result into a total lime replacement of 5,167 lbs of CaO for 200%, 3,194 lbs CaO for 100%, and 1,015 lb CaO for 50% design basis per day.

TABLE 10 Biological Oxygen Demand and Total Organic Carbon of 160 ºC Samples

Lysing Agent	Design Basis	BOD_5, mg/L	TOC, mg/L	Sample ID
NaOH	200%	37,000	42,500	080604-JH-10
NaOH	100%	37,000	38,300	080604-NB-05
NaOH	50%	26,000	26,800	080504-MV-06
H_2S0_4	100%	28,000	32,300	081104-JH-1
H_2S0_4	50%	24,000	24,600	081104-NB-1

TABLE 11 Titration of Basic Treated Sludge with WWT Influent and Acid Treated Sludge with Calcium Hydroxide Solution of 160ºC Samples

Lysing Agent	Design Basis	Titration Volume of Basic Treated Sludge*, mL	Calcium Oxide required **, g
NaOH	200%	0.30	-
NaOH	100%	0.517	-
NaOH	50%	1.60	-
H_2S0_4	100%	-	5.6.
H_2S0_4	50%	-	1.3

* Per 250 g of influent waste water at a pH of 4.82

** per 100 mL of Acidic Treated Sludge

The neutralization of acidic hydrolyzate results in a requirement of 5.6 grams of calcium oxide per 100 mL of hydrolyzate for 100% design basis and 1.3 grams per 100 mL of hydrolyzate for 50% design basis. This would translate to an additional requirement of 4,914 lbs of CaO for 100% and 1,141 lbs of CaO for 50% design basis per day.

2.7 CONCLUSIONS

Overall, 15 of the 18 experiments performed were within 5º C of the experimentally selected temperature, and 12 of them had a deviation less than 5ºC. The average residence time of the system was 9.9 minutes. The significant variables that impact the performance of the system to meet the temperature and residence time performance are the biosludge flow rates, the lysing agent flow rates, and the liquid level. The main variable that can shut down Unit BX is the reactor pressure. This can be a result of overfilling the reactor, trapping gas, or overheating the contents in the reactor. Through examination of the average operating conditions, it is clear that the system was very delicate and susceptible to upset from a number of factors. In addition, there is not a clear set of trends that lead to best performance of the system. Operation of Unit BX at present is more of an art than a science due to the number of factors that can destabilize the system and the very narrow turn down range of the system. For the sample results no distinct connection between the input of raw sludge and the operation of Unit BX can be deduced which would result in a predictable product with predictable total solids, fluid properties, or biological performance potential. Despite the erratic and unstable operation of the system, it is possible to convert a solid 15% biosludge into a fluid medium for

transport and other possible applications and to operate in a continuous manner. It is clear that there may be potential in regards to recycling the sodium hydroxide hydrolyzate to waste water treatment due to the offsetting of neutralization chemicals, that the treated hydrolyzate is nutrient-rich, and has potential to have water-like characteristics.

CHAPTER 3 EVALUATION OF A WASTEWATER TREATMENT SYSTEM WITH THE RECYCLE OF TREATED HYDROLYZATE

3.1 INTRODUCTON TO THE RECYCLE OF TREATED HYDROLYZATE TO WWT

The treated hydrolyzate from Unit BX is a nutrient-rich material that has potential for recycle to WWT to eliminate incineration. A model was developed (Plate 1) to determine the impact of recycling treated sludge to WWT based on tests data of the hydrolyzate. Plate 1 is an excel spreadsheet that will accompany this thesis. The following sections will show all derivations of the relationships and the details to the adaptation of a similar scaled up version of Unit BX process to a hypothetical WWT system that is Plate 1. Then outline a method for execution of Plate 1 to project the possible impacts of incorporating treated hydrolyzate to a WWT system. Two key variables have a tremendous impact on the economics of the WWT system with recycle. These variables are the design basis of the treated sludge, D_{BLA}, and, χ, the percent wet untreated biosludge sent away from WWT after recycle. The design basis is the amount of lysing agent that is added to the wet sludge prior to heat treatment. The percent of untreated biosludge sent away from WWT after recycle is the ratio of the biosludge sent away from the system and the total sludge discharged before recycle. The difference of this variable with 100% is the ratio of the sludge returned to recycle. All tests will be reflected in terms of D_{BLA} and $(1-\chi)$

An outline of all the constituents of Plate 1 that will be covered are: the process basis of the WWT system without recycle; the assumptions used for the Plate 1; a derivation of the base case model of the WWT system without recycle; and, an extensive derivation of the model incorporating the recycle of treated sludge. Then an overview of the analysis of the WWT system with recycle using Plate 1 will be covered.

3.2 PROCESS BASIS OF A WASTEWATER TREATMENT SYSTEM

Before creating any model of any system or process, it is necessary to have the following:

- a clear understanding of the fundamental underlying principles that will steer the process
- a process design of all standard inputs
- a clear and valid set of assumptions
- a sound set of relationships between various properties to have a closed and balanced system

The fundamental principle behind a material balance is shown in Equation 11.

$$Accumulation = In - Out + Generation - Consumption \quad \textbf{(11)}$$

Equation 11 is the equation of continuity. The first assumption to be made for a WWT system is that it will be operated at steady state. So, the relationship in Equation 11 can be simplified to Equation 12 by setting the accumulation to 0.

$$0 = In - Out + Generation - Consumption \quad \textbf{(12)}$$

The chemical equilibrium and rate of reactions will be neglected because they will be incorporated in terms of process yields. The relationship will be further simplified to Equation 13.

$$0 = In - Out \quad \textbf{(13)}$$

With Equation 13 in mind, the main focus of the model simplifies to the material entering the system equals the material leaving the system. Additionally, any complex relationship can be incorporated into the relationship with the use of a generic product yield. This will be covered in Section 3.3 after incorporating the recycle of biosludge into the material balances.

Table 12 is a summary of the inlet and outlet background data for the process basis. The data that comprises Table 12 comes from actual process operations for the inputs. The inputs are typical annual operating averages of the WWT system at Eastman Chemical Company, Kingsport, TN. The output values are targets that are typical limits for discharge of effluent water [17,18].

Table 13 is a compilation of additional miscellaneous variables that are needed to compliment Table 12. The derivations will following in the next section.

One important factor is the unit conversion factor, γ_{conv}, for converting the units of small concentrations of BOD, TOC, inerts, and the volumetric flow rate, ppm or mg/L, of the stream to units of lb/day.

$$\gamma_{conv} = 0.012016\, lb \cdot L \cdot \min(mg \cdot gal \cdot day)^{-1} \qquad \textbf{(14)}$$

This unit conversion factor is used strictly to keep the units of the formulas homogeneous. The conversion factor and its units is shown in Equation 14.

The Ultimate BOD and the 5-day BOD and their relationship were discussed in Chapter 2.5. The sludge yield with BOD is taken from Metcalf et. al [2]. The percentages of carbon and oxygen are taken from the accepted general molecular formula for biomass, $C_5H_7NO_2$ [2]. Lastly, the conversion factor of the ppm to lb/day is used for

TABLE 12 Background Process Data

In			Out		
Stream Property	**Average**	**Units**	**Stream Property**	**Average**	**Units**
BOD_{5-1} **	685	ppm	BOD_{5-4} ***	20	ppm
TOC_1 **	421.6	ppm	TOC_4 ***	10	ppm
$\dot{V}_1$ *	20,000	gpm	-	-	-
$Inerts_1$ **	65	ppm	-	-	-
$F_{Ca(OH)_2-1}$ **	6,500	tons/year	-	-	-

* The values are actual operating conditions

** The values are based off of annual averages

*** The values are based off of typical release limits and personnel experience

TABLE 13 Miscellaneous Properties of the Properties

Property Description	Property Nomenclature	Value	Units	Source
Weight % Concnetration of $Ca(OH)_2$ solution	$C_{Ca(OH)_2}$	20	%	[17,18]
Concentration of NaOH solution	C_{LA}*	50	%	-
Concentration of H_2SO_4 solution	C_{LA}**	98	%	-
Stream 1 Specific Gravity	σ_1	1	Unitless	Water
Stream 2 Specific Gravity	σ_2	1.1	Unitless	Measured
Stream 3 Specific Gravity	σ_3	1	Unitless	Water
Stream 4 Specific Gravity	σ_4	1	Unitless	Water
Biosludge Specific Gravity	σ_5	0.945	Unitless	[17,18]
Density of Water	ρ_{water}***	8.33	lb/gal	-
50% NaOH Solution Specific Gravity	σ_8*	1.52	unitless	-
98% H_2SO_4 Solution Specific Gravity	σ_8**	1.84	unitless	-
Ratio of BOD_U to BOD_5	$\nu_{\frac{BOD_U}{BOD_5}}$	70	%	[2,17,18]
Sludge Yield	$\Delta_s Y$	0.42	lb biomass /lb BOD_U	[2,17,18]
Percent Carbon in Biomass	λ_{Carbon}	~53.1	%	[2]
Percent Oxygen in Biomass	λ_{Oxygen}	~28.3	%	[2]
Percent Carbon in Carbon Dioxide	η_{Carbon}	~27.3	%	Calculated
Percent Oxygen in Carbon Dioxide	η_{Oxygen}	~72.7	%	Calculated

* Based on tests using sodium hydroxide
** Based on tests using sulfuric acid
*** The density of each stream is a product of the

conversion of the dilute concentrations into a useable form with the volumetric flow rate of a stream to determine the bulk mass flow rate in lb/day of a component in a stream.

The mass flow rate is assigned the variable F expressed in units of lb/day. The volumetric flow rate is assigned the variable $\dot{V}$ expressed in units of gal/day. Conversion between a mass flow rate and a volumetric flow rate of a stream is effected by dividing the variable (depending on which direction of conversion) by the density. In addition to variable assignment, there are subscripts that will be used to identify the item of a stream being manipulated (inerts, biomass, sludge, 5 day(5), ultimate (U) …) and/or the stream of the item is in (1,2,3 …). These subscripts will be ordered as mentioned.

The following sections will outline all the steps to create Plate 1. The process begins with the benchmark case of a WWT system without recycle. This will be the base case. Then further treatment of the derivation will include the recycle stream of the system.

3.3 CREATION OF A WASTEWATER TREATMENT SYSTEM WITH RECYCLE

To begin the evaluation of the WWT process with the recycle of biosludge, a rudimentary model of the WWT system is created. Figure 27 illustrates this model and the numbering of the corresponding streams. Table 14 summarizes the stream number and the general description of the streams contents. Streams 1 and 2 are the inlet streams. Streams 3, 4, and 5 are the outlet streams. Stream 1 is the wastewater that enters the WWT system. Stream 2 is the calcium hydroxide solution used to neutralize the inlet stream 1 to a pH of 7. The solution consists of a 20% solution of calcium hydroxide 80% water by mass. The average annual consumption at Eastman is 6,500 tons per year [18].

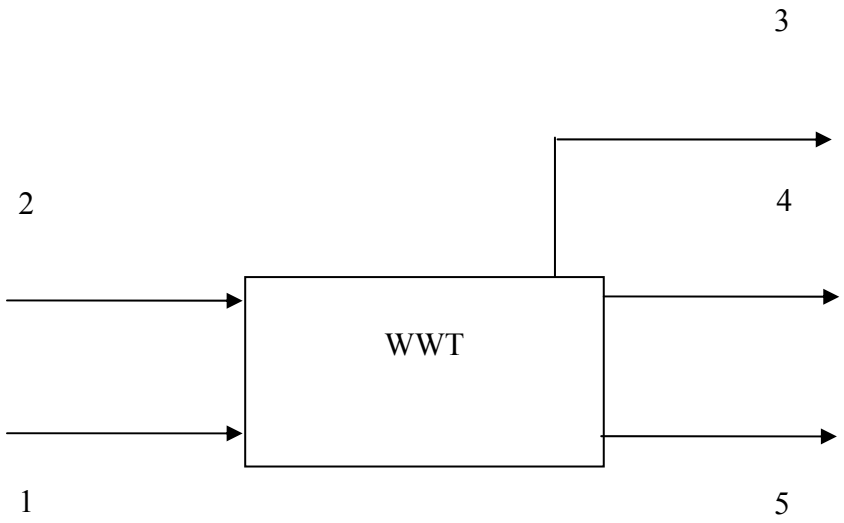


FIGURE 27 Wastewater Treatment System without Recycle Diagram

TABLE 14 Stream Identification Table for WWT without Recycle

Stream Number	Stream Description
1	Wastewater influent entering WWT
2	Calcium hydroxide solution for neutralization of low pH of Influent
3	Carbon dioxide produced from microorganisms consuming organic compounds in WWT influent
4	Cleaned water discharged from WWT
5	Biosludge solid waste stream

An assumption made with regard to this stream is that the calcium hydroxide will neutralize the acidity of the influent salt forming soluble salts that will be removed from the system by means of stream 4. Stream 3 consists of the carbon dioxide that is the byproduct of the biochemical reaction taking place inside the microorganisms in the waste water treatment system. The flow rate of stream 3 is an unknown and will be calculated by either the carbon balance or the oxygen balance used in the model. Stream 4 is the effluent water of the WWT system. The components in stream 4 are shown in Table 12. One assumption made for this stream is that all the inerts entering the waste water treatment system will settle and exit stream 5. The inerts will not be included in this stream. Stream 5 is the dewatered sludge stream. It consists of 85% water and 15% biosludge by mass. The biosludge consists of biomass and inerts. Typical operation of the WWT facility at Eastman produces approximately 50 tons/day of biosludge, dry basis. In addition to the production of biosludge, it is assumed that the average yield of biomass is 0.42 lbs biomass to 1 lb BOD_U. The chemical formula for biomass that will be used is $C_5H_7NO_2$. The percentage of carbon and oxygen in this formula is 53.1% and 27.3% respectively. With the layout of the details for each stream, construction of the material balances can be made.

Equation 15 outlines the generic carbon balance constructed.

$$F_{Carbon-1} + F_{Carbon-2} = F_{Carbon-3} + F_{Carbon-4} + F_{Carbon-5} \quad \textbf{(15)}$$

Since the carbon in stream 3 is unknown, the relationship will be rearranged as follows in Equation 16. Additionally, there is no carbon being added to the system via stream 2. So, stream 2 can be removed from the relationship.

$$F_{Carbon-1} - F_{Carbon-4} - F_{Carbon-5} = F_{Carbon-3} \quad \textbf{(16)}$$

The following equations will transform the generic terms into the nomenclature shown in Table 2, Equation 17, Equation 18, and Equation 19.

$$F_{Carbon-1} = \dot{V}_1 \gamma_{conv} TOC_1 \quad \textbf{(17)}$$

$$F_{Carbon-4} = \dot{V}_4 \gamma_{conv} TOC_4 \quad \textbf{(18)}$$

$$F_{Carbon-5} = BOD_{5-1} \nu_{\frac{BOD_U}{BOD_5}} \dot{V}_1 \gamma_{conv} (1 - \beta_{inerts}) \lambda_{carbon} \quad \textbf{(19)}$$

Where β_{inerts} is the factor of inerts present in the dry biosludge. Equation 20 shows the relationship of the inerts with the biosludge, β_{inerts}.

$$\beta_{inerts} = \frac{F_{inerts-5}}{F_{biosludge-5}} \quad \textbf{(20)}$$

Where

$$F_{inerts-5} = F_{inerts-1} = \dot{V}_1 \gamma_{conv} \quad \textbf{(21)}$$

And

$$F_{sludge-5} = F_5 \theta_5 = F_{biomass-5} + F_{Inerts-5} \quad \textbf{(22)}$$

Equation 23 combines all of the elements shown in Equations 17 – 20 and substitutes these to create the total carbon balance.

$$\begin{aligned} &\dot{V}_1 \cdot \gamma_{conv} \cdot TOC_1 - \dot{V}_4 \cdot \gamma_{conv} \cdot TOC_4 \\ &\quad - \dot{V}_1 BOD_{5-1} \cdot \gamma_{conv} \cdot \nu_{BODU/BOD5} \cdot \Delta_S Y (1 - \beta_{Inerts}) \cdot \lambda_{carbon} = F_{Carbon-3} \end{aligned} \quad \textbf{(23)}$$

The mass flow rate of the carbon dioxide exiting the system can be determined by using Equation 24.

$$F_{CO_2} = \frac{F_{Carbon-5}}{\eta_{carbon}} \quad \textbf{(24)}$$

The same procedure will be followed for the oxygen balance. However, the BOD_5 of each stream and the oxygen content of the biomass will be used instead of TOC. A similar relationship can be made for the oxygen balance by using Equation 16 and using notation to implicitly specify oxygen instead of carbon, shown in Equation 25.

$$F_{Oxygen-1} - F_{Oxygen-4} - F_{Oxygen-5} = F_{Oxygen-3} \quad \textbf{(25)}$$

Where

$$F_{Oxygen-1} = BOD_{5-1}\nu_{\frac{BOD_U}{BOD_5}}\dot{V}_1\gamma_{conv} \quad \textbf{(26)}$$

$$F_{Oxygen-4} = BOD_{5-4}\nu_{\frac{BOD_U}{BOD_5}}\dot{V}_4\gamma_{conv} \quad \textbf{(27)}$$

$$F_{Oxygen-5} = BOD_{5-1}\nu_{\frac{BOD_U}{BOD_5}}\dot{V}_1\gamma_{conv}(1-\beta_{inerts})\lambda_{Oxygen} \quad \textbf{(28)}$$

These terms can be again substituted into Equation 25 to yield Equation 29

$$BOD_{5-1}\nu_{\frac{BOD_U}{BOD_5}}\dot{V}_1\gamma_{conv} - BOD_{5-4}\nu_{\frac{BOD_U}{BOD_5}}\dot{V}_4\gamma_{conv} - BOD_{5-1}\nu_{\frac{BOD_U}{BOD_5}}\dot{V}_1\gamma_{conv}(1-\beta_{inerts})\lambda_{Oxygen} = F_{Oxygen-3} \quad \textbf{(29)}$$

Where the flow rate of stream 3 can be determined by using a similar relation shown in Equation 30.

$$F_{CO_2} = \frac{F_{Oxygen-5}}{\eta_{Oxygen}} \quad \textbf{(30)}$$

These are the underpinning relationships used in the WWT model without recycle. The closure of the balances can be determined by the agreement of the carbon dioxide flows based from each balance pathway. The model with recycle is the base case that is used. In later discussions, the model will return to the aforementioned relationships when the recycle split ratio is set to 1. The following section will cover the steps to creation of the WWT model with a recycle loop included.

3.3 CREATION OF A WASTEWATER TREATMENT SYSTEM MODEL WITH RECYCLE

Figure 28 illustrates the stream relationship of the WWT system with recycle. It is an extension of the model shown in Figure 27, where the box labeled I is the equivalent model without recycle and the box labeled II is the model used to factor in the recycle of treated hydrolyzate.

Table 15 outlines all of the stream descriptions for Figure 28. The simplest approach to begin derivation of all the relationships of the flow rates of the streams of a WWT system with a recycle stream of treated sludge is a balance of the inerts flowing through the WWT system. This approach will elicit an understanding of the flow atterns in the recycle stream with a slipstream of sludge leaving the WWT system to be treated and recycled to the inlet of the WWT system.

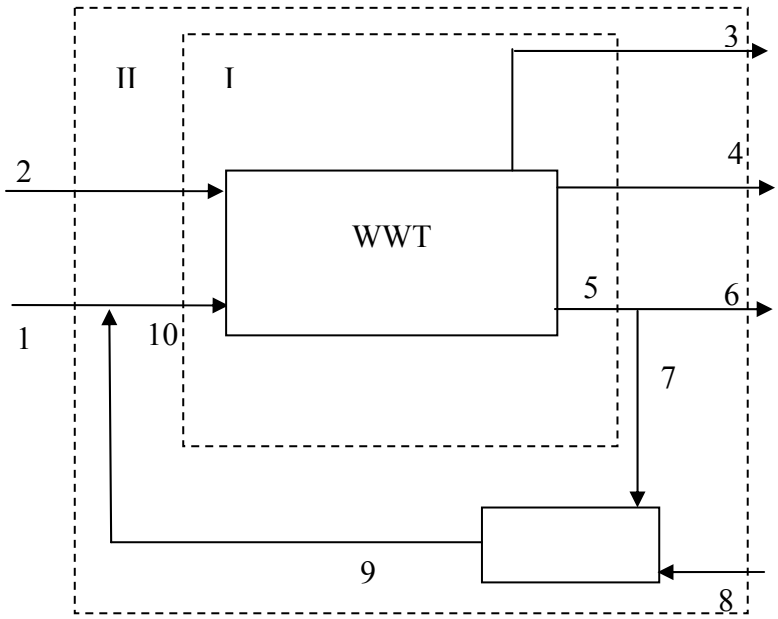


FIGURE 28 Material Balance Diagram for WWT System with Recycle.

TABLE 15 Stream Identification Table for WWT with Recycle

Stream Number	Stream Description
1	Wastewater influent entering WWT before hydrolyzate stream
2	Calcium hydroxide solution for neutralization of low pH of Influent
3	Carbon dioxide produced from microorganisms consuming organic compounds in WWT influent
4	Cleaned water discharged from WWT
5	Biosludge solid waste stream discharged from WWT before recycle
6	Biosludge solid waste stream after recycle split stream
7	Biosludge solid waste stream that is being sent to recycle
8	Lysing agent added to biosludge recycle stream
9	Treated hydrolyzate product stream
10	Wastewater influent stream and hydrolyzate product stream entering WWT System

The variable used to express the flow rate of the slipstream relative to the total wet sludge flow rate leaving the waste water system prior to the split of exiting sludge and slipstream is χ. χ will have a range greater than 0 but less than 1. (1- χ) is the ratio of the flow rate of the wet sludge slip stream with respect to the flow rate of the sludge slip stream. Equations 31 and 32 illustrate the relationship of χ and the two streams

$$\chi = \frac{F_6}{F_5} \quad \textbf{(31)}$$

$$(1-\chi) = \frac{F_7}{F_5} \quad \textbf{(32)}$$

An overall material balance of the inerts will be performed to first define two key assumptions, the inerts will only enter in the influent stream 1 and be purged from the system in the exit stream 6, Equation 33 illustrates this relationship. Additionally the same phenomena will be true for the inlet stream 10 to the WWT system and the exit stream 5 prior to the exit of the slip stream as shown in Equation 34.

$$F_{1-Inerts} = F_{6-Inerts} \quad \textbf{(33)}$$

$$F_{10-Inerts} = F_{5-Inerts} \quad \textbf{(34)}$$

Further development of stream 5 and 6 is shown in Equation 35. The relationship that compliments the relationship between stream 5 and 7 shown in Equation 36.

$$F_{6-Inerts} = F_{5-Inerts}\chi \quad \textbf{(35)}$$

$$F_{7-Inerts} = F_{5-Inerts}(1-\chi) \quad \textbf{(36)}$$

A material balance of the inlet stream 1 and stream 9 resulting in stream 10 shown in Equation 37,

$$F_{1-Inerts} + F_{9-Inerts} = F_{10-Inerts} \quad \textbf{(37)}$$

Since it is assumed that inerts will not be added to the recycle stream via stream 8 the flow rate of inerts of stream 7 equals the flow rate of inerts in stream 9, Equation 38.

$$F_{7-Inerts} = F_{9-Inerts} \quad \textbf{(38)}$$

Substitution of Equations 34, 36, 38 into 37 yield Equation 39.

$$F_{5-Inerts} = F_{1-Inerts} + F_{5-Inerts}(1-\chi) \quad \textbf{(39)}$$

Solving for the inerts in stream 5 yield Equation 40.

$$F_{5-Inerts} = \frac{F_{1-Inerts}}{\chi} \quad \textbf{(40)}$$

The result of this derivation is that the internal flow rates are a direct function of the inlet flow rate and the ratio of the inerts leaving the system and the flow rate of the inerts prior to being split for recycle. Equation 41 further clarifies the details of the total inlet volumetric flow rate of stream 1, the concentration of the inerts, and a conversion factor, to provide a more convenient relationship to use in further derivations and calculations.

$$F_{5-Inerts} = \frac{\dot{V}_1 I \gamma_{Conv.}}{\chi} \quad \textbf{(41)}$$

The relationship of the the flow rate of stream 5 inerts could further be expanded to include the biomass of stream 5 since the total flow rate is a linear combination of the inerts and the biomass.

To begin discussion of the derivation of the total flow rate, the relationship shown in Equation 38 will be expanded to include the flow rate of the lysing agent and everything else that will constitute stream 7, Equation 42.

$$F_9 = F_8 + F_7 \tag{42}$$

Equation 43 is a volumetric expression of 42.

$$\dot{V}_9 = \frac{F_8}{\rho_8} + \frac{F_7}{\rho_7} = \dot{V}_8 + \dot{V}_7 \tag{43}$$

Equation 36 can be expanded in a similar manner for wet sludge as shown in Equation 44.

$$F_7 = F_{5-wet\,sludge}(1-\chi) = \frac{F_{5-sludge}(1-\chi)}{\theta} \tag{44}$$

Where θ is the percent dried solids contained in wet sludge. It is necessary to differentiate the qualities of wet sludge, dry sludge, biomass, and inerts. The dried sludge is the combination of biomass and inerts in the stream. This relationship for stream 7 is illustrated in Equation 45.

$$F_5 = F_{5-wet\,sludge} = \frac{(F_{5-sludge})}{\theta} = \frac{(F_{5-biomass} + F_{5-inerts})}{\theta} \tag{45}$$

Streams 5, 6, and 7 are wet sludge, for this matter the notation of wet sludge in the subscript will be left out with the understanding that this is the case for these specific streams.

Subsitution of Equations 41 and 45 into Equation 44 results in Equation 46.

$$F_7 = \frac{(F_{5-biomass} + F_{5-inerts})(1-\chi)}{\theta} = F_{5-biomass}\frac{(1-\chi)}{\theta} + \dot{V}_1 I \gamma_{conv}\frac{(1-\chi)}{\theta\chi} \quad \textbf{(46)}$$

Equation 46 illustrates the relationship of the inlet concentration and the flow rate of the biomass coming from stream 5. Equation 47 includes the density of the biomass of the sludge to yield a volumetric flow rate of stream 7,

$$\dot{V}_7 = F_{5-biomass}\frac{(1-\chi)}{\theta\rho_7} + \dot{V}_1 I \gamma_{conv}\frac{(1-\chi)}{\theta\rho_7\chi} \quad \textbf{(47)}$$

where the density of the sludge in stream 7 is the same as the density in stream 5 and stream 6 as shown in Equation 48.

$$\rho_5 = \rho_7 = \rho_6 \quad \textbf{(48)}$$

The density of stream 5 will be used since all resulting derivations will be based on the flow rate of the biomass.

Substitution of Equation 48 into Equation 47 yields Equation 49.

$$\dot{V}_7 = \frac{F_{5-biomass}(1-\chi)}{\theta\rho_5} + \frac{F_{5-inerts}(1-\chi)}{\theta\rho_5} \quad \textbf{(49)}$$

The flow rate of the lysing agent is a function of the flow rate of stream 7. The relationship was illustrated in Equations 2 and 3. These relationships are expanded to show the relationship with the inert and biomass flow rates of stream 7. Equation 50 illustrates the composition of this relationship.

$$F_8 = K_{LA}F_{7-wet\ sludge} = \frac{K_{LA}F_7}{\theta} = \frac{K_{LA}F_{5-biomass}(1-\chi)}{\theta} + K_{LA}\dot{V}_1 I \gamma_{conv}\frac{(1-\chi)}{\theta\chi} \quad \textbf{(50)}$$

Equation 51 expands this relationship further.

$$F_8 = F_{5-biomass}(1-\chi)\frac{\eta_{LA}D_B}{C_{LA}} + \dot{V}_1 I \gamma_{conv}\frac{(1-\chi)}{\chi}\frac{\eta_{LA}D_B}{C_{LA}} \quad \textbf{(51)}$$

Equation 52 is the volumetric expression of Equation 51 of the lysing agent solution.

$$\dot{V}_8 = F_{5-biomass}(1-\chi)\frac{\eta_{LA}D_B}{\rho_8 C_{LA}} + \dot{V}_1 I\gamma_{conv}\frac{(1-\chi)}{\chi}\frac{\eta_{LA}D_B}{\rho_8 C_{LA}} \quad \textbf{(52)}$$

Equations 52 and 49 are substituted into Equation 43 to yield Equation 53.

$$\dot{V}_9 = F_{5-biomass}(1-\chi)\left(\frac{1}{\theta\rho_5}+\frac{\eta D_B}{C_{LA}\rho_8}\right) + \dot{V}_1 I\gamma_{conv}\frac{(1-\chi)}{\chi}\left(\frac{1}{\theta\rho_5}+\frac{\eta D_B}{C_{LA}\rho_8}\right) \quad \textbf{(53)}$$

Equation 54 is used to further simplify the relationship shown in Equation 53 and simplify further derivations.

$$\Gamma = \left(\frac{1}{\theta\rho_5}+\frac{\eta D_B}{C_{LA}\rho_8}\right) \quad \textbf{(54)}$$

Equation 55 illustrates the simplification of equation 53.

$$\dot{V}_9 = F_{5-biomass}(1-\chi)\Gamma + \dot{V}_1 I\gamma_{conv}\frac{(1-\chi)}{\chi}\Gamma \quad \textbf{(55)}$$

The relationships of the inlet stream 1 and stream 9 equal stream 10 for the inerts, Equation 57.

$$F_1 + F_9 = F_{10} \quad \textbf{(56)}$$

Equation 57 is the volumetric representation of Equation 56.

$$\dot{V}_1 + \dot{V}_9 = \dot{V}_{10} \quad \textbf{(57)}$$

During the derivation of the inerts balance in Equation 57, the inerts in stream 10 was equal to the inerts in stream 5; however, this is not the case for the biomass. The biomass in the synthesis of the material balances is based on a yield of biomass relative to an amount of BOD_U. Equations 58 and 59 illustrate the variables that will be used for these numerical terms and units.

$$\Delta_s Y = \frac{0.42 lb\ Dry\ Biomass}{1 lb\ BOD_U} \quad \textbf{(58)}$$

$$\nu_{\frac{BOD_U}{BOD_5}} = \frac{1 lb BOD_U}{0.7 lb BOD_5} \quad \textbf{(59)}$$

These factors combined with the conversion factor γ_{conv} are illustrated in Equation 60.

$$\psi = \Delta_s Y \gamma_{conv} \nu_{\frac{BodU}{Bod5}} \quad \textbf{(60)}$$

Equation 61 is a combination of Equations 55, 57, 50, and the BOD5 of each stream to solve for the biomass flow rate of stream 5.

$$F_{5-biomass} = \dot{V}_1 \psi Bod_{5-1} + F_{5-biomass}(1-\chi)\Gamma \psi Bod_{5-9} + \dot{V}_1 \psi I \gamma_{conv} \frac{(1-\chi)}{\chi} Bod_{5-9} \quad \textbf{(61)}$$

Solving for the biomass of stream 5 yields Equation 62.

$$F_{5-biomass} = \frac{\dot{V}_1 \psi \left(Bod_{5-1} + I\gamma_{conv} \frac{(1-\chi)}{\chi} Bod_{5-9} \right)}{\left(1-(1-\chi)\Gamma Bod_{5-9}\psi\right)} \quad \textbf{(62)}$$

Equation 62 is a key relationship since it is only a function of the volumetric flow rate of stream 1, the BOD of the treated sludge, the concentration of the inerts, the split ratio and the design basis.

All other stream relationships can be created from this relationship as shown in Equations 63, 64, 65, 66, 67, and 68.

$$\dot{V}_9 = \dot{V}_1 (1-\chi)\Gamma \left(\frac{\psi \left(Bod_{5-1} + I\gamma_{conv} \frac{(1-\chi)}{\chi} Bod_{5-9} \right)}{\left(1-(1-\chi)\Gamma Bod_{5-9}\psi\right)} + \frac{I\gamma_{conv}}{\chi} \right) \quad \textbf{(63)}$$

$$\dot{V}_{10} = \dot{V}_1\left(1+(1-\chi)\Gamma\left(\frac{\psi\left(Bod_{5-1}+I\gamma_{conv}\frac{(1-\chi)}{\chi}Bod_{5-9}\right)}{\left(1-(1-\chi)\Gamma Bod_{5-9}\psi\right)}+\frac{I\gamma_{conv}}{\chi}\right)\right) \quad \textbf{(64)}$$

$$\dot{V}_8 = \dot{V}_1(1-\chi)\frac{\eta_{LA}D_B}{C_{LA}\rho_8}\left(\frac{\psi\left(Bod_{5-1}+I\gamma_{conv}\frac{(1-\chi)}{\chi}Bod_{5-9}\right)}{\left(1-(1-\chi)\Gamma Bod_{5-9}\psi\right)}+\frac{I\gamma_{conv}}{\chi}\right) \quad \textbf{(65)}$$

$$\dot{V}_7 = \frac{\dot{V}_1(1-\chi)}{\theta\rho_5}\left(\frac{\psi\left(Bod_{5-1}+I\gamma_{conv}\frac{(1-\chi)}{\chi}Bod_{5-9}\right)}{\left(1-(1-\chi)\Gamma Bod_{5-9}\psi\right)}+\frac{I\gamma_{conv}}{\chi}\right) \quad \textbf{(66)}$$

$$\dot{V}_5 = \frac{\dot{V}_1}{\theta\rho_5}\left(\frac{\psi\left(Bod_{5-1}+I\gamma_{conv}\frac{(1-\chi)}{\chi}Bod_{5-9}\right)}{\left(1-(1-\chi)\Gamma Bod_{5-9}\psi\right)}+\frac{I\gamma_{conv}}{\chi}\right) \quad \textbf{(67)}$$

$$\dot{V}_6 = \frac{\dot{V}_1\chi}{\theta\rho_5}\left(\frac{\psi\left(Bod_{5-1}+I\gamma_{conv}\frac{(1-\chi)}{\chi}Bod_{5-9}\right)}{\left(1-(1-\chi)\Gamma Bod_{5-9}\psi\right)}+\frac{I\gamma_{conv}}{\chi}\right) \quad \textbf{(68)}$$

The flow rate of stream 2 is based on the average usage of calcium hydroxide in an annual period as shown in Equation 69.

$$F_{2-Calcium\ Hydroxide} = 6500\frac{tons}{year} \quad \textbf{(69)}$$

Equation 70 is a conversion to lbs/day usage and 71 is the total flow rate of a 20% solution of calcium hydroxide.

$$F_{2-Calcium\ Hydroxide} = \frac{6500\,ton}{1\,year}\left|\frac{1\,year}{365\,days}\right|\frac{2000\,lbs}{1\,ton} = 35{,}616.44\,\frac{lbs}{day} \qquad \textbf{(70)}$$

$$F_2 = 178{,}082.19\,\frac{lbs}{day} \qquad \textbf{(71)}$$

Equation 72 is a conversion of the mass flow rate to a volumetric flow rate.

$$\dot{V}_2 = \frac{F_2}{\rho_2} \qquad \textbf{(72)}$$

Equation 75 is the expression of the liquid flow rate of the liquid stream 4. Please note that the solution of stream 2 titrates with the influent but no mass is added to stream 5 as a result. This is idealized because the concentration of calcium in the sludge could not be quantified at the time of analysis.

$$\dot{V}_4 = \dot{V}_1\left(1 - \frac{(1-\theta)}{\theta\rho_5}\left(\frac{\psi\left(Bod_{5-1} + I\gamma_{conv}\dfrac{(1-\chi)}{\chi}Bod_{5-9}\right)}{\left(1-(1-\chi)\Gamma Bod_{5-9}\psi\right)} + \frac{I\gamma_{conv}}{\chi}\right)\right) + \dot{V}_2 \qquad \textbf{(73)}$$

As was derived earlier with the WWT system without recycle, a carbon and oxygen balance are used to determine the flow rate of carbon dioxide leaving in stream 3. The same will be performed for the WWT system with recycle. The difference is the TOC and BOD_5 of the recycled biomass will be included in the equations. Equation 74 will express the carbon balance for all streams entering and exiting the system.

$$\begin{aligned} &TOC_{10}\cdot\dot{V}_{10}\cdot\gamma_{conv} - TOC_4\cdot\dot{V}_4\cdot\gamma_{conv} \\ &\quad -\dot{V}_1\gamma_{conv}BOD_{5-10}\cdot\nu_{\frac{BOD_U}{BOD_5}}\cdot\Delta_S Y\left(1-\beta_{Inerts}\right)\cdot\lambda_{carbon} = F_{TIC-3} \end{aligned} \qquad \textbf{(74)}$$

There is little difference between Equation 74 and the carbon balance for the WWT system without recycle other than incorporating the carbon load from the recycle loop. Equation 77 illustrates the changes for the oxygen balance.

$$BOD_{5-10} \cdot \nu_{\frac{BOD_U}{BOD_5}} \cdot F_{10} \cdot \gamma_{conv} - BOD_{5-4} \cdot \nu_{\frac{BOD_U}{BOD_5}} \cdot F_4 \cdot \gamma_{conv} - \dot{V}_1 BOD_{5-10} \cdot \nu_{\frac{BOD_U}{BOD_5}} \lambda_{oxygen} \cdot \Delta_S Y(1-\beta_{Inerts}) \cdot \gamma_{conv} = F_{TIC-3}\left(\frac{1}{\eta_{Carbon}} - 1\right) \quad \textbf{(75)}$$

With Equations 74 and 75 the flow rate of carbon dioxide leaving the system

$$F_3 = F_{TIC-3}\left(\frac{1}{\eta_{Carbon}}\right) \quad \textbf{(76)}$$

Additionally, the $BOD_{5\text{-}10}$ is shown in Equation 77.

$$BOD_{5-10} = \frac{BOD_{5-1}F_1 + BOD_{5-9}F_9}{F_1 + F_9} \quad \textbf{(77)}$$

3.5 RECYCLE OF TREATED BIOSLUDGE TO WWT MODEL EXPERIMENTAL METHODOLOGY

The model of the WWT system with recycle of treated biosludge, Plate 1, is a method of projecting the impacts of recycling a nutrient rich material into WWT. The methodology for examining the impacts will consist of defining the metrics for examination, defining the key variables of the system, and specifying the procedures used to obtain data for the examination of the impacts. The key variables are the lysing agent used, the percentage of design basis of the lysing agent, and the choice percentage of untreated biosludge recycled to the WWT system. The metrics are the mass flow rate of the untreated wet biosludge discharged from the WWT system, calcium hydroxide consumption, lysing agent consumption, the mass flow rate of wet biosludge discharged

before the recycle discharge split, the biological oxygen demand load, the total organic carbon load, and the mass flow rate of carbon dioxide generated. In addition, a comparison of the effects with the absence of the inerts will be made for every metric using 200% and 100% sodium hydroxide lysing agent.

The metrics of the WWT system with recycle are direct products of derivations of Plate 1. The metrics are presented in units of tons/day. Table 16 shows the metrics and the corresponding mathematical relationships of the model. The representations shown in Table 16 are from the derivations discussed in Section 3.4 of the WWT system with recycle. The metrics are flow rates of corresponding streams shown in Figure 28. The untreated wet biosludge sent to disposal is the mass flow rate of stream 6. The reduction of $Ca(OH)_2$ is the mass flow rate of the dry lime minus the amount of lime that would be replaced or needed due to the introduction of the treated biosludge into the influent stream.

TABLE 16 The Metrics and Their Corresponding Mathematical Relationships to Figure 28

Metric	Mathematical Representation	Units
Untreated Sludge to Disposal	F_6	Tons/day
Reduction of $Ca(OH)_2$ Consumption	$F_2\theta_2 - F_9\xi$	Tons/day
Lysing Agent Consumption	F_8	Tons/day
Wet Sludge Discharge Before Recycle	F_5	Tons/day
Biological Oxygen Demand Load	$BOD_{5-10} = \frac{BOD_{5-1}\dot{V}_1\gamma_{conv} + BOD_{5-9}\dot{V}_9\gamma_{conv}}{\left(\dot{V}_1 + \dot{V}_9\right)}$	Tons/day
Total Organic Carbon Load	$TOC_{10} = \frac{TOC_1\dot{V}_1\gamma_{conv} + TOC_9\dot{V}_9\gamma_{conv}}{\left(\dot{V}_1 + \dot{V}_9\right)}$	Tons/day
Carbon Dioxide Generation	F_3	Tons/day

The lysing agent consumption metric is the mass flow rate of the lysing agent for stream 8. The wet sludge discharged before the recycle discharge recycle split is the mass flow rate of stream 5. The BOD load is the mass flow rate of the BOD into the WWT system via stream 10. The TOC load is the mass flow rate of TOC load entering the WWT system via stream 10. The carbon dioxide generation is the mass flow rate of the relative carbon dioxide that is created by the WWT system from the total organic carbon load and BOD entering the WWT of the system exiting via stream 3. These metrics are monitored with the variation of the three key variables and plotted against the amount of biosludge recycled. The figures will be shown in the results and discussion.

The key variables are the main factors that were shown in earlier studies at the University of Tennessee-Knoxville that drive the economics of the system [16]. These variables are constrained by a range for testing. The range for lysing agent selection is constrained by two primary options, sodium hydroxide and sulfuric acid. The percentage of design basis for a lysing agent is the percentage of the ratio of the lysing agent to dried biosludge used. For sodium hydroxide, these are 50, 100 and 200%. For sulfuric acid the percentages are 50 and 100%. The percentage of biosludge returned to recycle is the split discharge to recycle ratio. The range of the percentage recycle variable of the biosludge is from 0% to 100% being the maximum. In these studies a maximum of 90% will be used for the reason that the model will go to infinity as a result of the presence of inerts.

The experimental analysis of the model is performed by adjusting the key variables in Plate 1 and recording the values of the streams and/or the loads as shown in Table 17. Entry of the key variables will result in simultaneous solution of all the

properties. Table 14 outlines details to the experiments that will be performed using plate 1.

3.6 RECYCLE OF TREATED BIOSLUDGE TO A WWT RESULTS AND DISCUSSION

The WWT model (Plate 1) is a method of projecting the effects of recycling treated biosludge through a set of metrics by adjusting key variables that affect the operation economics of this treatment option. The key variables were the lysing agent used, the percentage of design basis, and the percentage of untreated wet biosludge recycled. The metrics are the mass flow rate of untreated biosludge discharged from the WWT system, calcium hydroxide consumption, lysing agent consumption, the mass flow rate of wet sludge discharge before the recycle discharge split, the BOD load, the TOC Load, and the carbon dioxide generated. In addition a comparison of the effects without inerts was made for every metric with respect to 200% and 100% design basis of sodium hydroxide lysing agent.

Figure 29 illustrates the discharge of wet untreated biosludge from WWT. In the range of recycle of 0% and 90%, the reduction of wet untreated biosludge discharge has a linear stoichiometric relationship.

TABLE 17 Experimental Outline for Tests with Plate 1

Lysing Agent	Design Basis	Percent Recycle	Percent Recycle Increments
Sodium Hydroxide with Inerts	50%, 100%, and 200%	0%-90%	10%
Sodium Hydroxide without Inerts	100% and 200%	0%-90%	10%
Sulfuric Acid	50% and 100%	0%-90%	10%

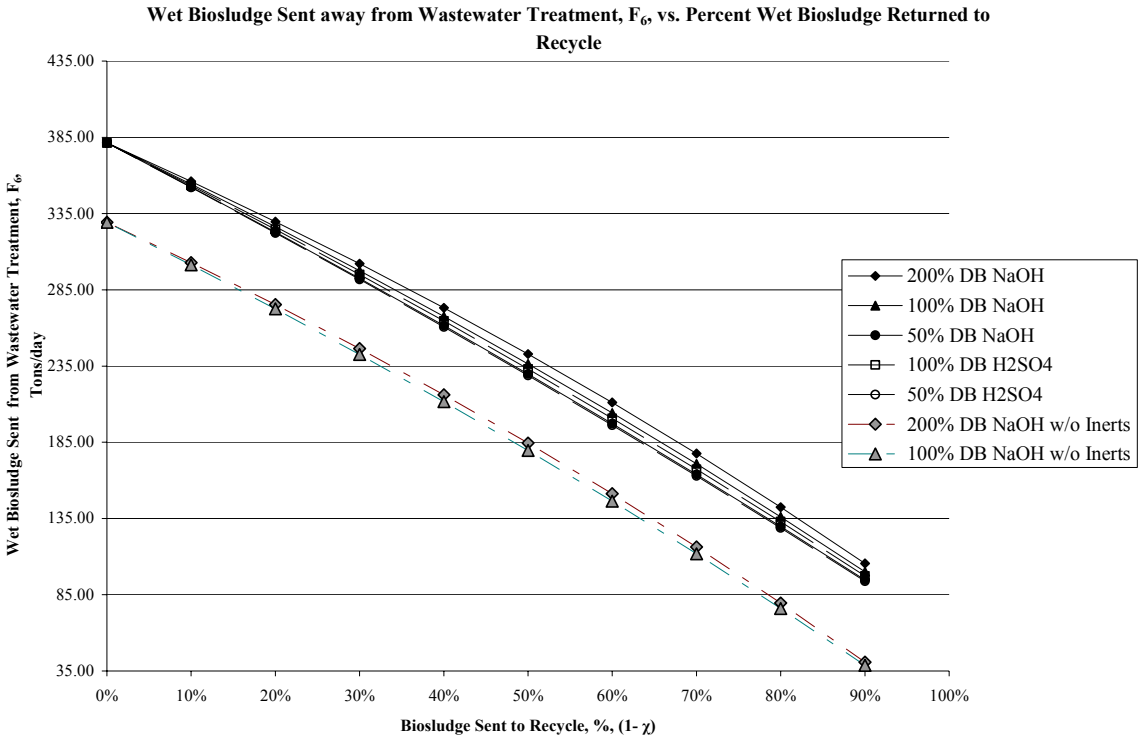


FIGURE 29 Wet Biosludge Sent Away from WWT with Respect to the Percentage of Wet Untreated Biosludge Sent to Recycle.

Between the different lysing agents, 200% sodium hydroxide design basis will result in less reduction of untreated biosludge than the 50% sodium hydroxide design basis. There is less reduction since the treatment of the biosludge at 200% design basis releases more nutrients than the 50% design basis. In turn, the microorganisms consume the nutrients, creating more biomass and retarding the elimination of the biosludge.

It appears to be beneficial to have a lower concentration lysing agent for treatment as opposed to having a strong treatment of the wet untreated sludge. Sulfuric acid in both 100% design basis and 50% design basis appears to have a more attractive result compared to sodium hydroxide. In addition, two tests were performed with a system without inerts for sodium hydroxide lysing agent at 200% and 100% design basis. The

proportional difference between the system with and the system without inerts is approximately 50 tons/day of wet untreated sludge. The apparent advantage of removing inerts from the system is that a hypothetical zero solids emissions could be achieved; where this would not be the case if the inerts were to remain in the system. The inerts would require a purge stream to minimize the recycle load such that there would not be an infinite buildup in the WWT system.

Figure 30 illustrates the reduction of calcium hydroxide with the percentage of untreated sludge sent to recycle. As seen, the trend for the increase of recycle for 200% sodium hydroxide design basis reduces the total requirement of calcium hydroxide consumption by 50% at 90% recycle of wet untreated biosludge. 100% sodium

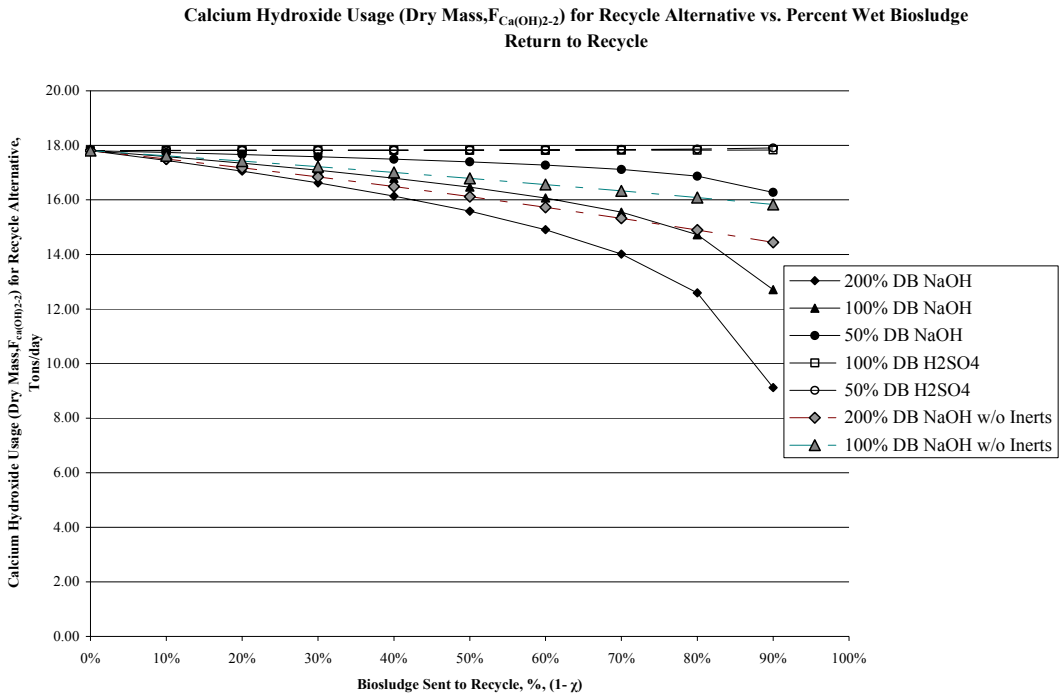


FIGURE 30 Calcium Hydroxide Consumption with Respect to the Percentage of Wet Untreated Biosludge Recycled to WWT

hydroxide design basis reduces the consumption by 25% at 90% recycle of wet untreated biosludge. Both 100% and 50% sulfuric acid design basis have little effect on the consumption of calcium hydroxide. The sodium hydroxide design basis for the system without inerts shows that the reduction is proportional to the percentage of untreated wet sludge recycled. Additionally, the difference of the two cases of the system with inerts and the system without inerts shows that the inerts further increase the reduction of calcium hydroxide due to their presence. The inerts are the cause of the large decrease, since they would be treated similarly as the biomass. Overall the higher concentration of sodium hydroxide results in the most reduction of consumption calcium hydroxide.

Figure 31 illustrates the consumption of lysing agents with respect to the amount of sludge sent to recycle. As shown, 200% sodium hydroxide design basis would have the highest consumption at 200 tons/day with inerts and 50% sulfuric acid design basis would have the least consumption at approximately 20 tons/day. Both lysing agents are expensive compared to calcium hydroxide shown in previous studies at the University of Tennessee-Knoxville[16]. Again, the presence of inerts further increases the operation and places the cost further beyond original estimates placing a negative light on the option at the initial stages of testing.

Figure 32 shows the total wet sludge discharge from the waste treatment system with respect to the percentage of untreated sludge sent to recycle. Clearly, it can be seen that the amount of sludge in the system with inerts hyperbolically increases with the percentage returned to recycle, suggesting that the quantity of sludge would rise

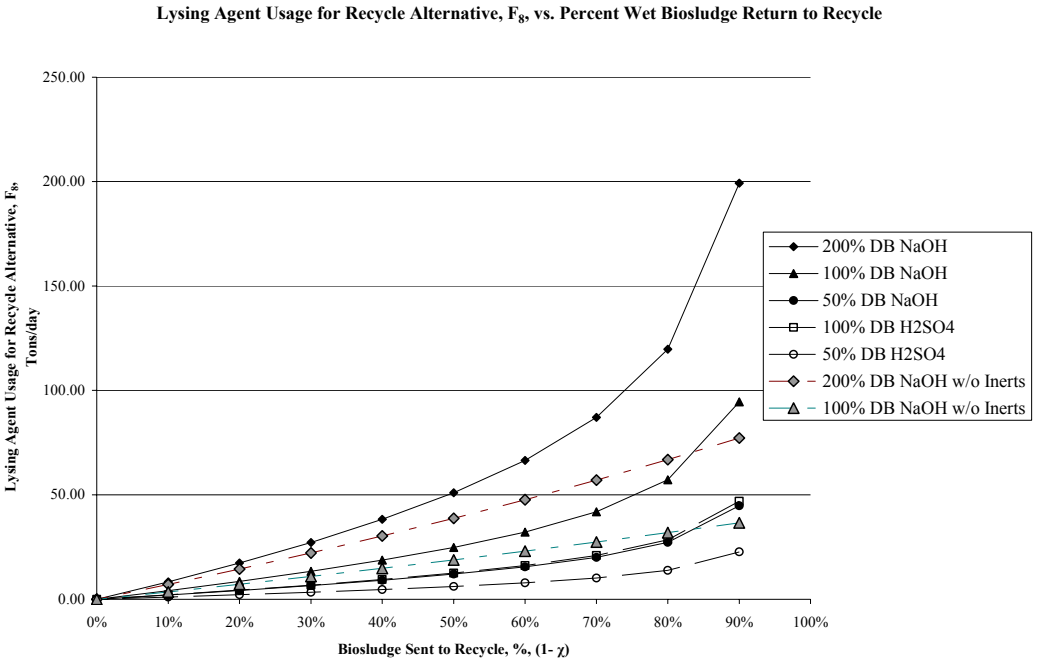


FIGURE 31 Lysing Agent Usage for Recycle Alternative with Respect to the Percentage of Untreated Biosludge Returned to Recycle.

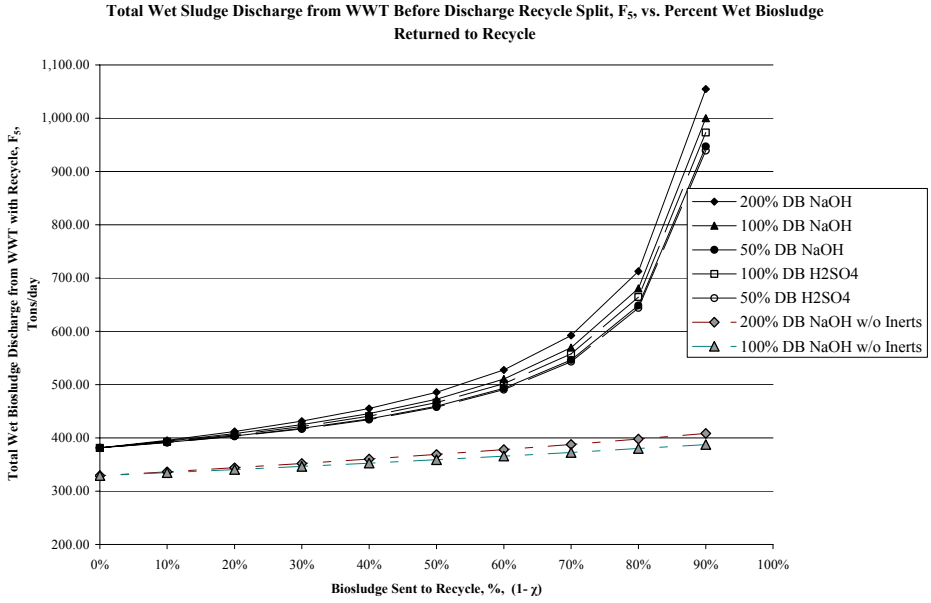


FIGURE 32 Total Wet Sludge Discharge from WWT Before Discharge Recycle Split with Respect to the Percentage of Wet Untreated Sludge Sent to Recycle.

indefinitely as the percentage of wet untreated biosludge sent to recycle approaches 100%; whereas, the system without inerts rises linearly, suggesting a possibility of complete recycle of the sludge and that the maximum of this option would result in a recycle stream of 100 tons/day at 100% recycle.

Figure 33 illustrates the BOD of the inlet feed stream 10 that enters the WWT system with respect to the percentage of sludge that is sent to recycle. Again, a similar trend occurs. Recycle with the inerts results in a higher nutrient flow rates resulting in a huge increase in the BOD load on the WWT. This would present a problem with most aerobic WWT systems in that they are designed with a maximum load due to aeration requirements being limited by aeration capabilities. This would be a negative feature for consideration. The WWT system at Eastman that this model was designed around has a maximum BOD load of 200,000 lbs/day. The recycle of treated sludge with 200% sodium hydroxide design basis would reach this maximum at approximately 60% recycle of sludge; whereas, at 100% sodium hydroxide design basis would be at approximately 70% recycle. The 50% sulfuric design basis would reach the limit at approximately 85%. If the inerts were removed, potentially 100% recycle could be achieved for recycle with the lower sodium hydroxide design basis and sulfuric acid design basis. Overall, the 50% sulfuric acid design basis appears to be the most promising selection.

Figure 34 illustrates the TOC load on the WWT system with respect to the recycle of treated biosludge to the WWT. A similar trend occurs as with the BOD; however, since there are no set restrictions or bottlenecks for this metric, the trend is meant to show the relationship of TOC with the percent recycle.

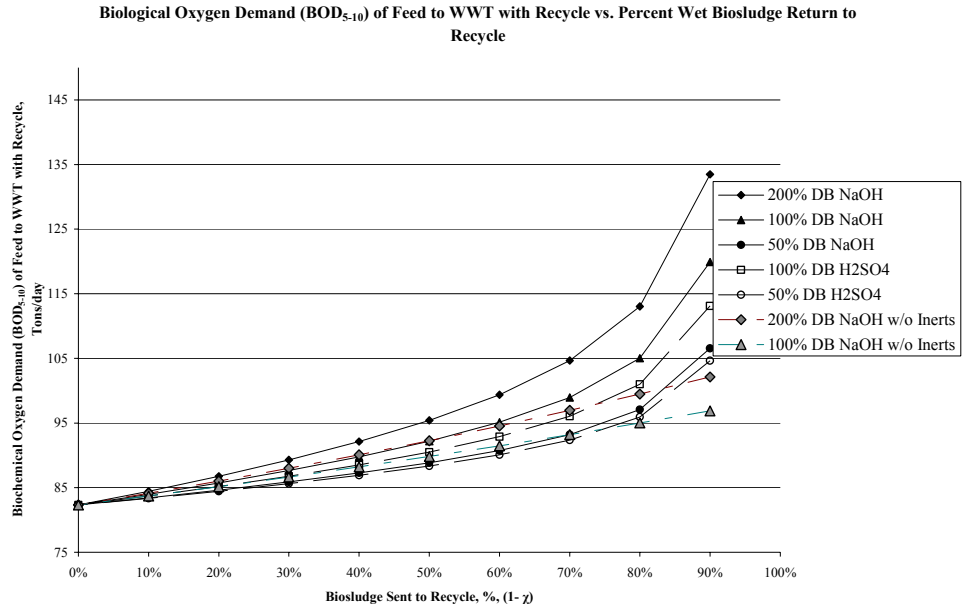


FIGURE 33 BOD of the Inlet Feed Stream(BOD_{5-10}) of WWT with Respect to the Percentage of Recycle of Wet Untreated Biosludge.

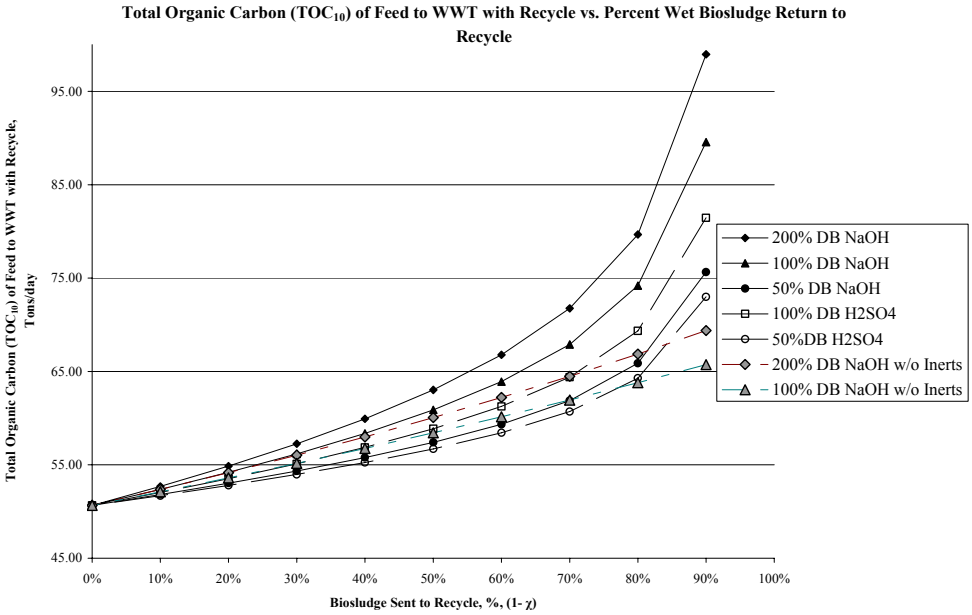


FIGURE 34 TOC of the Inlet Feed Stream (TOC_{10}) of WWT with Respect to the Percentage of Recycle of Wet Untreated Biosludge.

The trend shows the hyperbolic relationship of the TOC with the percent recycle and the linear relationship of the system without inerts.

Figure 35 illustrates the carbon dioxide generation from biological oxidation of the nutrients in the WWT system. Overall, the maximum potential for carbon dioxide generation is the system without inerts. The lowest potential for carbon dioxide generation is the system with 200% sodium hydroxide design basis. For the systems with inerts, the maximum potential could be reached for 50% sulfuric acid design basis.

3.7 CONCLUSIONS

The treated biosludge is a nutrient-rich medium that can be recycled into the WWT system and be consumed to create carbon dioxide, reducing the total output of untreated wet biosludge, thus reducing the consumption of calcium hydroxide

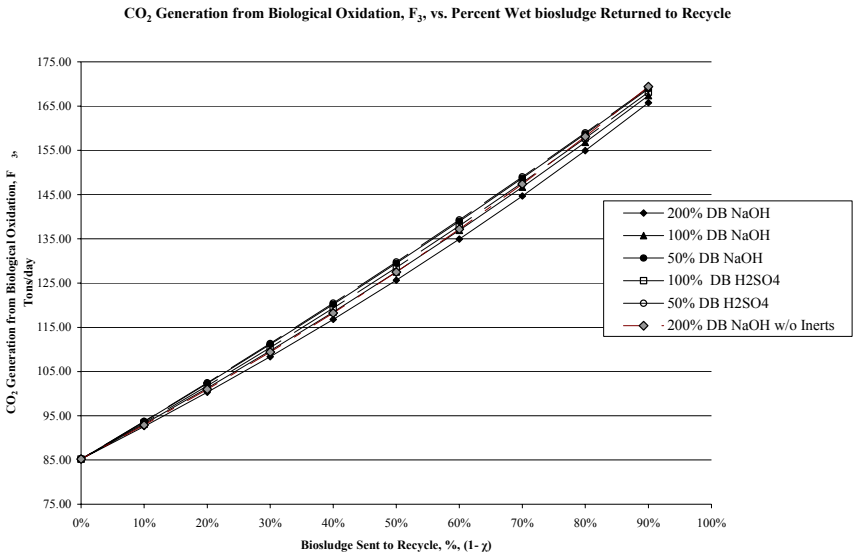


FIGURE 35 Carbon Dioxide Generation from Biological Oxidation with Respect to Percent of Untreated Biosludge Returned to WWT.

However, in contrast, this option consumes more expensive lysing agent raw materials, has potential to create an enormous recycle stream, and can overload the biological treating capacity of the WWT. In addition, the presence of inerts in the system would further increase the negative impact of this option. This study has shown that the optimal lysing agent for recycle of treated biosludge is 50% sulfuric acid design basis in terms of waste reduction and loading requirements. The worst selection is 200% sodium hydroxide design basis in terms of raw material consumption and overall loading requirements. The optimal design basis of both lysing agents is 50% due to the minimal impacts. A system with the recycle of treated sludge that operates without inerts would be the choice for zero solid waste emissions.

CHAPTER 4 SLURRYING HYDROLYZATE WITH COAL

4.1 INTRODUCTION

The biosludge hydrolyzate is an energy rich material that contains carbon and hydrogen and has a water-like consistency. The biomass that composes approximately 15% of the hydrolyzate has approximately 53% carbon and 6% hydrogen. The remaining 85% of the hydrolyzate is water. Commercial coal slurry gasification processes rely on carbon and hydrogen from coal and water to slurry the coal to generate carbon monoxide and hydrogen. With the chemical composition and the water-like consistency, the hydrolyzate is an attractive potential alternative to plain water used in commercial coal slurry gasification processes. In addition to the potential to slurry coal with hydrolyzate, converting the biosludge waste stream into a raw material stream for other chemical products and a renewable energy resource would prove to be environmentally and economically beneficial.

The first step in applying biosludge hydrolyzate to a commercial coal slurry gasification process will require addressing the impact on the physical transport properties of the hydrolyzate coal slurry. The goal is to make coal slurries with the partial addition of hydrolyzed biosludge and compare the physical properties to a standard coal slurry mixture. This will be accomplished by defining a standard coal slurry for comparison, adapting a mixture of the treated sludge to match measurable gravitometric characteristics of a the standard coal slurry, defining the method of creating a coal slurry, creating grind samples of both standard coal and water/hydrolyzate coal slurries, measuring the physical properties of the coal slurry samples (total solids, pH, coal

particle size distribution, viscosity, and foaming tendency), and making comparisons of the results between the standard and water/hydrolyzate coal slurries.

4.2 STANDARD COAL SLURRY MIXTURE

The standard coal grind mixture consists of a combination of coal, water, and particle suspending surfactant Ammonium Lignin Sulfonate (ALS). This mixture is used to assay various qualities of different sources of coal to assist in decision making concerning what treatments would be necessary to the coal prior to allowing the coal to being incorporated into a gasification process. Additionally, this mixture is used to assay the slurriability of the coal. This mixture is designed to match typical operating conditions of a commercial gasification process sized for laboratory testing. The mixture consists of coal, water, and ALS in the following mass proportions 850g, 400g, 2.5g respectively. This mixture is used as a reference for comparison in further studies. The ALS is an optional component dependant on the characteristics of the coal and its pH. The application of ALS with a treated sludge/water coal slurry will be discussed further as an optional component in the following section.

The water/biosludge hydrolyzate coal grind mixture closely resembles the standard coal grind mixture. The formulation is based on two principles. First, the solids content of the slurry is equivalent to the solids content of a typical standard coal slurry mixture. Second, the mixture matches the production volume of the usage of coal on a daily basis and the production of biomass. In addition, the water is used to make up the difference of mass used. The key difference between the two mixtures is the use of ALS since the application of ALS is pH-dependant. Since the possibility exists that foaming of

the slurry shown from pilot studies performed at Eastman with a different treated biosludge, a defoaming agent will be applied to minimize the chances of foaming during the study.

The following section is a complete derivation of the mixture that will be used for this study. The analysis will be used for every treated biosludge sample tested.

4.3 HYDROLYZATE/WATER COAL SLURRY MIXTURE

The mixture for the standard coal grind consists of the following weight percent proportions ~68% Solids, ~32% water, and ~<1% of ALS. The function of the ALS in the coal slurry is to enhance the wettability of the coal allowing for suspension of the coal particles in water. The pH range for effective use of ALS is 5-7 [19]. The pH ranges of the treated hydrolyzate and coal mixtures are either greater than 10 or less than 1. The use of ALS may not be an appropriate additive. Pilot coal grind studies with the treated sludge were performed without the application of ALS. Findings from the studies show that the suspension of coal solids was superior compared to ALS as shown in Figure 36. The left-hand picture shows the standard coal slurry with ALS. It appears to have settled as indicated by the level of water on top of the coal solids. However, the picture on the right shows the treated biosludge with the coal grind forming a smooth and creamy appearance, even after being allowed to sit in a sealed container for two weeks. As a result, the ALS was left out of the treated biosludge/water coal slurry mixture. The primary mixture uses the same distribution of liquid and solids as the standard coal slurry. The next step in the development of the mixture was to assess what production level hydrolyzate quantity to slurry with coal could be achieved.

FIGURE 36 Left Standard Coal Slurry Sample Allowed to Settle; Right Treated Sludge Coal Slurry Allowed to Settle

At Eastman, an average usage of 100 tons of coal per hour is used. This is divided by the total solids of the slurry resulting in the total slurry mass flow rate as shown in Equation 78.

$$\frac{F_{Coal}}{\theta_{Slurry\ Solids}} = F_{Total\ Slurry} \qquad \textbf{(78)}$$

The difference of the mass flow rate of the total slurry and the coal yields the mass flow rate of the water required to slurry with the coal as shown in Equation 79.

$$F_{SlurryWater} = F_{Total\,Slurry} - F_{Coal} \qquad \textbf{(79)}$$

The next step is to determine the amount of water present in a treated sludge on a production scale. The average production of untreated biosludge at Eastman is 50 tons wet sludge per day. Since the addition of lysing agent changes the liquid and solids content of the biosludge, this was taken into account. So to incorporate this into the analysis, the average total solids of a sample set of treated sludge was used in conjunction with the flow rate of the biosludge to ascertain the flow rate of water a lysing treatment

process. The average total solids of the sample set that is run at 140°C were used. This value is ~16.3% the value of the water flow rate is shown in Equation 80.

$$F_{Hydrolyzate} - F_{Hydrolyzate}\theta_{Hydrolyzate\ Solids} = F_{Hydrolyzate\ Water} \quad \textbf{(80)}$$

A ratio of the amount of water from the treated sludge and the total water required is created as shown in Equation 81.

$$\theta_{Hydrolyzate\ Water} = \frac{F_{Hydrolyzate\ Water}}{F_{Total\ Slurry\ Water}} \quad \textbf{(81)}$$

It turns out the that the operating examples used at Eastman result in ratio of ~50% treated sludge water and ~50% fresh water.

With this in mind, the treated slurry has a percentage of solid associated with the adaptation of the treated sludge. Since the constraint of the total solids of the coal slurry is ~68%, the coal will be reduced to accommodate for this adjustment. Equation 82 is used to determine the solids that would be present in the hydrolyzate to accommodate 200 grams of hydrolyzate water.

$$X = F_{Hydrolyzate\ Solids} = \frac{\theta_{Hydrolyzate\ Solids}}{1-\theta_{Hydrolyzate\ Solids}} F_{Hydrolyzate\ Solids} \quad \textbf{(82)}$$

The mixture for the hydrolyzate/water coal slurry is 200g treated sludge water, 200g water, X grams of solids from the treated sludge, and 850g-X of coal.

4.4 COAL SLURRY

Regardless or whether the coal slurry is plain or contains biomass, the preparation and slurry creation process with respect to grinding the coal with the appropriate additives are the same. The as-received coal is a moist substance with large chunks of coal, various sizes of medium sized coal, and coal dust. For laboratory experimentation,

a homogeneous coal sample was used. The focus was the medium sized coal particles. The process began with receipt of the wet raw coal sample as shown in Figure 37. The coal was placed in open plastic bins. Shallow 2 ft x 2.5 ft x 0.5 ft bins are found to be optimal for laboratory scale production. The moist coal was evenly spread into the bin and allowed to air dry overnight. The coal was then separated into specific sizes. Sieve mesh sizes 6 and 16 were used to separate coal particles from chunks of coal that are larger than size 6 and coal fines and dust smaller than sieve size 16. The sieve size 6 is 6 square channels per square inch. The sieve size 16 is 16 square channels per square inch. The coal that is greater than size 6 and less than size 16 was discarded.

The material between sieves 6 and 16was collected and appropriately stored. Figure 38 shows the homogenous material that was used in the experiments.

FIGURE 37 As-Received Unsieved Moist Coal

FIGURE 38 Air Dried 6-16 Mesh Sieved Coal

The appropriate amounts of coal, water, and additional additives were added to the ball grinding mill. The empty ball grinding mill with the grinding balls is shown in Figure 39. The contents of the mixture was added after the balls to avoid any splashing and potential material loss.

The ball-grinding mill was then sealed and placed on a ball mill tumbler for slurry processing. With the laboratory equipment used, it was possible to run two tests in the laboratory (as shown in Figure 40). The ball mill was allowed to tumble for 90 minutes. After the 90 minutes have elapsed, the ball mill(s) was (were) removed from the tumbler and further processed.

FIGURE 39 Ceramic Balls in Ball Grinding Mill

FIGURE 40 Ball Mill Grinding Tumbler

The tumbling crock was slowly opened to allow the escape of trapped/generated gases. Figure 41 shows the opened vessel and slurry/ball mixture. The slurry and ceramic balls were then removed from the grinding crock. Care was taken to remove all slurry debris. In some circumstances, the material caked on the sides of the vessel and in crevices, preventing a homogeneous grind. This debris was accounted for in following tests. The balls and slurry were separated using a course mesh size. A size 4 mesh sieve worked acceptably since the slurry particles were a smaller than the mesh dimensions (as shown in Figure 42).

The slurry sample was collected in a storage vessel for a brief period of time while all grinding equipment wass cleaned for the next use.

Three aliquots of the slurry samples were removed for measuring the total solids of the slurry. The same procedure was followed that was used for measuring the total solids of the treated biosludge discussed in chapter 2.51.

After measuring the total solids of the slurry, the viscosity was measured using a viscometer. Figure 43 shows the viscometer used for coal slurry samples.

Then the coal slurry's pH was measured followed by a particle size distribution analysis. Further examination of the sample could be an ash fusion test or a qualitative observation of the settling of the solids over an extended duration.

Recipes for the typical standard coal slurry and the slurry for sludge adaptation will be discussed in detail in the following sections. The analysis procedures will be covered in detail in the experimental section

FIGURE 41 Coal Slurry and Ball Grinding Ball Mixture Prior to Extraction

FIGURE 42 Coal Slurry Extraction

FIGURE 43 Brookfield DVII+ Viscometer

4.5 EXPERIMENTAL COAL SLURRY MEASUREMENTS

4.5.1 Experimental Introduction

The experimental portion of the coal slurry testing focused on the details of analysis of the slurry samples. The measurements performed were the total solids, the pH, the viscosity, and the particle size distribution of the coal slurries.

4.5.2 Total Solids Slurry Measurement

The process to obtain total solids of a coal slurry was performed in three steps: initial weighing of a wet coal slurry sample, drying the sample for a period overnight or for 24 hours, and weighing the dried coal slurry sample.

The first step was to take three aliquots of well mixed coal slurry samples and place in a tared thermally stable drying container. The mass of the tare and the wet sample were weighed on an analytical balance. The aliquots of samples were placed in a drying oven maintained at 104 ^{o}C for a period of 24 hours. The samples were removed from the oven and allowed to cool to room temperature. After cooling, the samples were weighed on an analytical balance. The difference of the mass before and after drying the total moisture removed from the sample. The ratio of the mass of moisture removed and the mass of the wet sample is the weight percent moisture. One hundred percent minus the weight percent of the moisture of the sample is the weight percent of solids in a sample. This test was performed three times to obtain an average and a standard deviation. The average is the reference of the total solids in all of the coal slurry.

4.5.3 Coal Slurry pH measurement

The method for measuring pH was described in Chapter 2.54. The difference was that the pH of the coal slurry was measured instead of treated hydrolyzate. The overview of the process for the measurement that was used well-mixed sample is placed into a container. A calibrated pH probe was placed into the sample. And, the measurement was taken. The probe was cleaned and put away for used at another time.

4.5.4 Coal Slurry Viscosity Measurement

The measurement of viscosity was described in Chapter 2.5.5. The viscosity of the coal slurry was measured in a similar method. However, only 2 spindles were used, LV-2 and LV-3. Since the coal slurry was a non-homogenous mixture, only two measurements of viscosity were made, the initial viscosity and the final viscosity. The

spindle was placed into the well-mixed coal slurry and a measurement was taken. After a short period of time, the final viscosity was measured. The spindle, speed, percent torque, and the viscosity were recorded.

4.5.5 Coal Slurry Particle Size Distribution

Particle size distribution of the coal slurry consists of taking a 100 g sample of the the coal slurry and washing the slurry through a set of clean preweighed sieves. The sieves sizes were 20, 40, 100, 200, and 325 mesh sizes. The mesh size is the number of holes per square inch.

100 g of well mixed coal slurry sample was weighed and placed into a tared 250 mL beaker. The sample was placed into the top of the stacked set of sieves, 20 mesh size at the top of the stack, 325 at the bottom. The slurry was then washed with tap water to rinse away the residual liquid and the smaller. After adequate washing of each sieve, the samples were dried for a minimum of 24 hours and all evidence of moisture was no longer present. The sieves with the coal fines were then weighed. The difference of the mass of the final weight of each sieve and the empty sieve wass calculated and recorded.

4.6 COAL SLURRY RESULTS AND DISCUSSION

The treated hydrolyzate is an energy-rich medium with a water like consistency with potential to slurry with coal. Comparisons have been made with the standard coal grinds. The metrics of comparison of the coal grind are total solids, pH, viscosity, and particle size distribution of the coals slurries. Emphasis has been placed on samples that closely resembled properties of the standard slurries. The summary of the samples

properties have been displayed in a similar way as the summaries of the treated hydrolyzate shown it Chapter 2.6. The Averages of the samples properties are shown with respect to the samples experimental range. In the charts, the standard coal slurries properties shown first, the sodium hydroxide hydrolyzate coal slurries shown in the middle, and the sulfuric acid coal slurries shown at the end of the chart.

Figure 44 illustrates the average total solids of the slurries with respect to the experimental test range. The average coal grind was within a few percent of 68%. Coal grinds that had a total solids percentage greater than 70% were 75% design basis at 150ºC for sodium hydroxide and 50% and 100% design basis for sulfuric acid. The grinds that had an average performance less than 68% was the 100% design basis at 170°C for sodium hydroxide.

Figure 45 show the total solids content of samples that are the within the standard deviation of the standard coal grind. The sample sets that closely resembled the standard coal grind in total solids were 50%, 75%, and 200% at 140ºC, 75%, 100%, and 200% at 160ºC, and 50% and 200% at 170ºC for sodium hydroxide lysing agent.

Figure 46 illustrates the pH of the coal slurries with respect to the experimental test range. The pH of the standard coal slurries was approximately 2.8 shown to the far left of the graph. For the sodium hydroxide samples starting at a high design basis, the 200% samples were close to a pH of 14. As the design basis for the sodium hydroxide samples were reduced, the pH approached 10. In addition to the reduction of the pH, the deviations in the sample pH increased at lower design basis. For the sulfuric acid samples, the pH at 100% design basis is less then 1. As the design basis for the sulfuric acid was reduced the pH increased. In both cases for the sulfuric acid and sodium

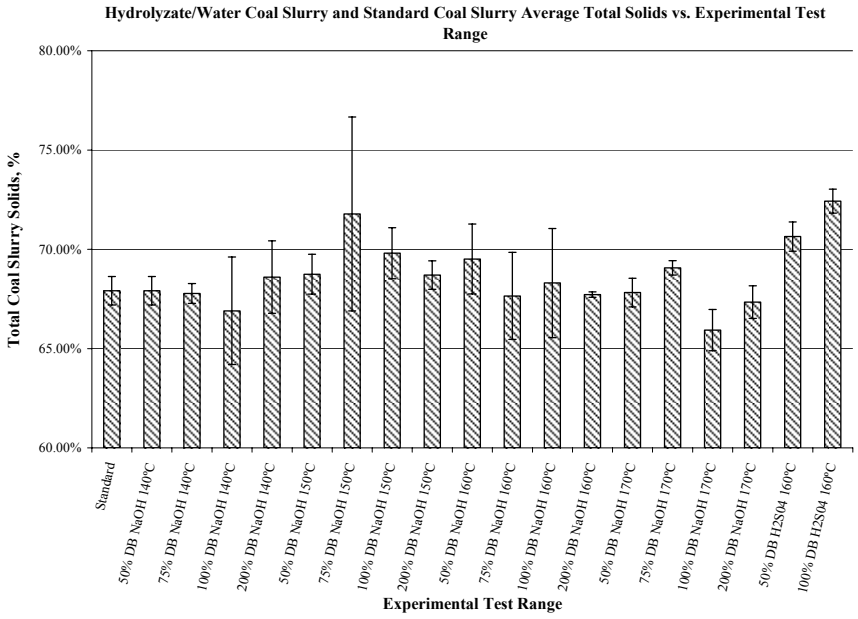


FIGURE 44 Hydrolyzate/Water Coal Slurry and Standard Coal Slurry Average Total Solids with Respect to Experimental Test Range

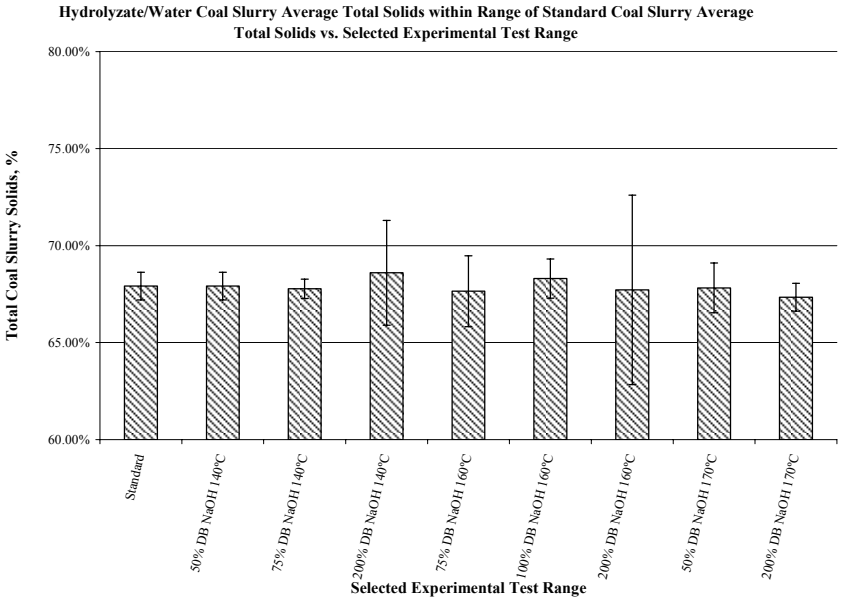


FIGURE 45 Hydrolyzate/Water Coal Slurry Average Total Solids within Range of Standard Coal Slurry Average with Respect to Selected Experimental Test Range

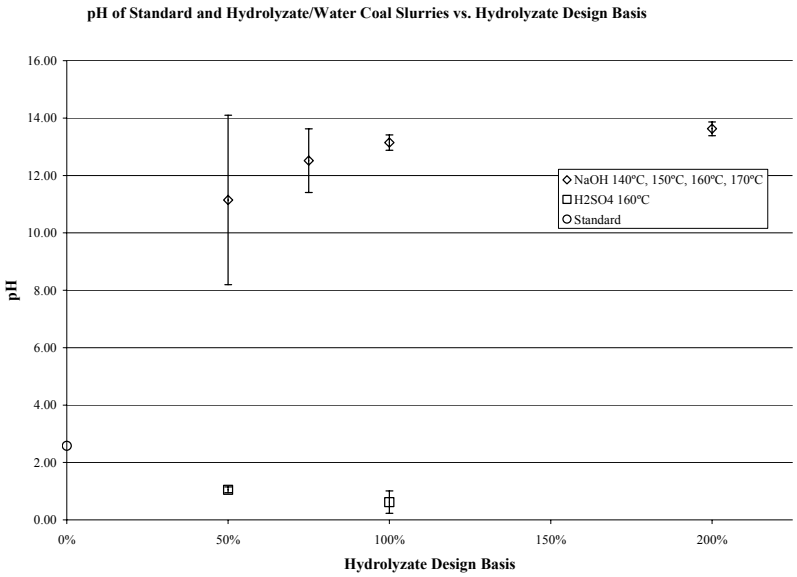


FIGURE 46 pH of Standard and Hydrolyzate/Water Coal Slurry Coal Slurries with respect to Hydrolyzate Design Basis

hydroxide, the pH of the sample approached the pH of the standard coal slurry.

In addition, for the sodium hydroxide samples, the relative pH of the coal in general was more acidic than the hydrolyzate sodium hydroxide samples and more basic than the sulfuric acid samples.

Figure 47 illustrates the average initial and final viscosities of all of the coal slurries with respect to the experimental test range. The viscosity for the standard coal slurries was less than 5,000 cP. The samples that had an extraordinarily high viscosity range greater than 40,000 cP were the low temperature, low design basis for the sodium hydroxide lysing agent. The viscosity of the 50% and 75% at 140ºC samples were unmeasurable. For the sulfuric acid samples, the 50% design basis had an extremely high

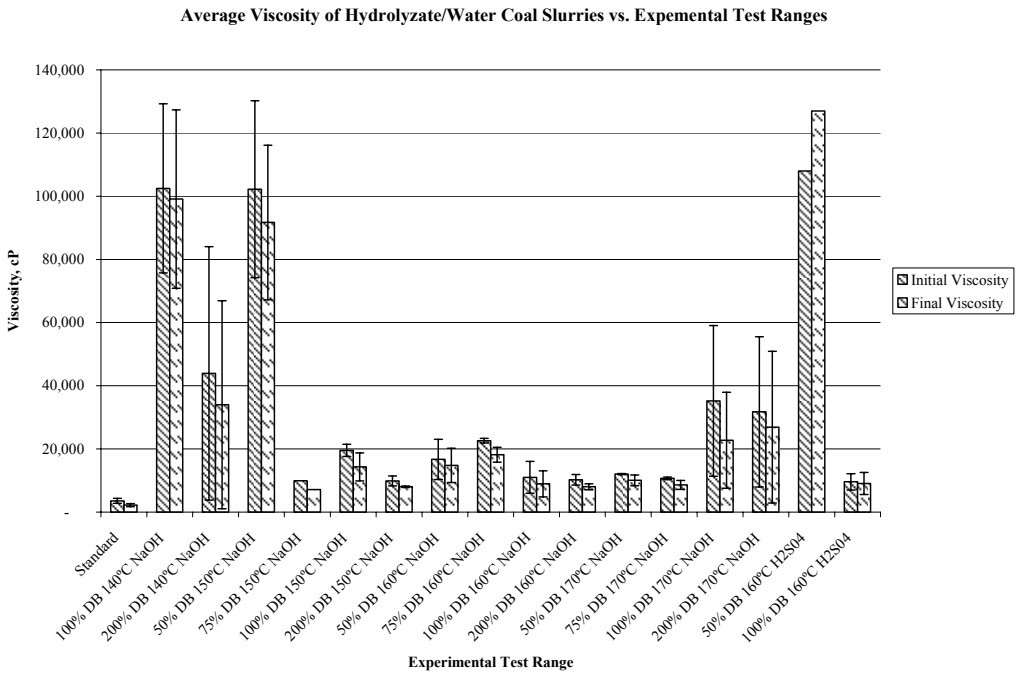


FIGURE 47 Average Viscosity of Hydrolyzate/Water Coal Slurries with Respect to Hydrolyzate Experimental Test Range

viscosity. The occasional tendency of these samples to foam increased the viscosity This was seen with the samples at lower temperature and lower design bases.

Figure 48 is a graph similar to figure 49, with the exception that the focus was placed on samples with a viscosity less than 25,000 cP. This graph eliminates all of the extremely high viscosity samples and shows that there is potential that the hydrolyzate can closely resemble the viscosity of the standard coal slurry. The better performing samples were in the 150-160 degree temperature range and elevated lysing agent. Figure 49 is a similar depiction with the maximum average viscosity no greater than the 12,000 cP. The better performing samples are in the 150-160 temperature range.

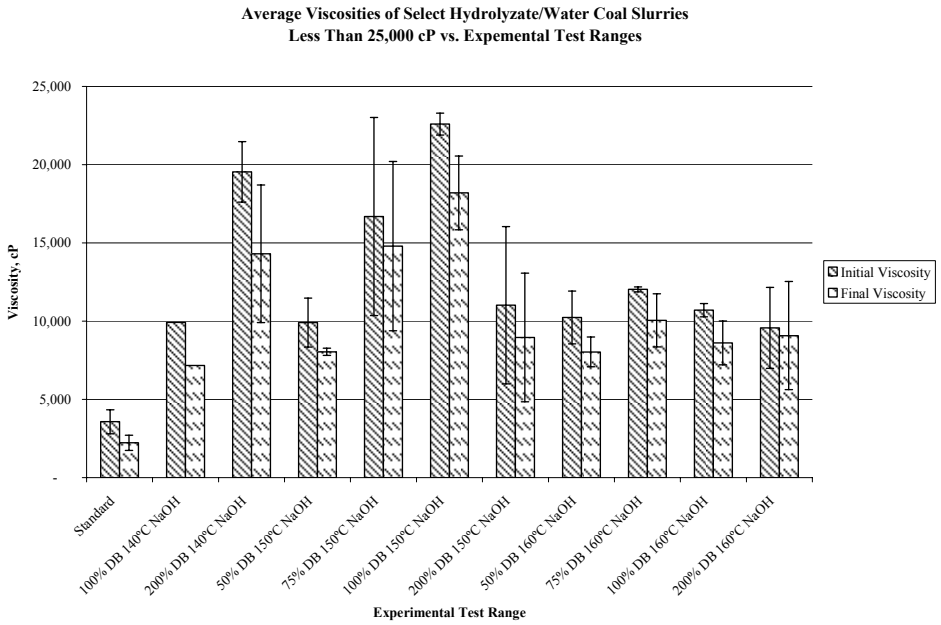


FIGURE 48 Average Viscosity of Select Hydrolyzate/Water Coal Slurries Less 25,000 cP with Respect to Hydrolyzate Experimental Test Range

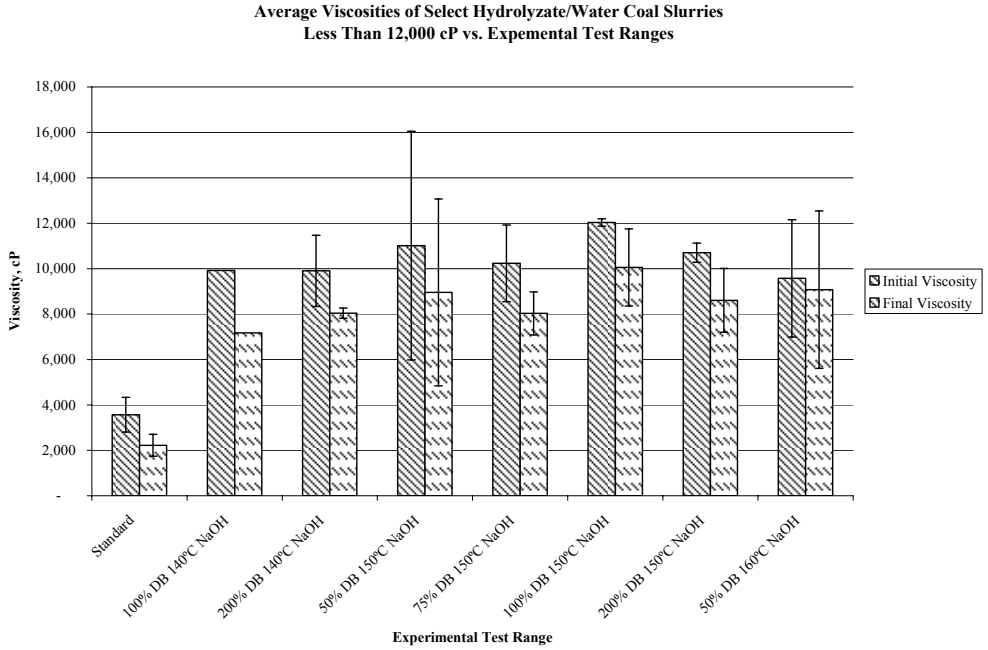


FIGURE 49 Average Viscosities of select Hydrolyzate/Water Coal Slurries Less Than 12,000 cP with respect to Hydrolyzate Experimental Test range

Even though the hydrolyzate/water grinds performed very similarly to each other, the viscosities were more than twice the viscosity of the standard coal grind. This suggests that the hydrolyzate may be pumpable, but with difficulty. One phenomenon that was noticed and suggested in the preparation of the coal grinds was that the coal slurry blend for some of the samples had a creamy appearance. In addition to having this appearance, when the slurry samples with the hydrolyzate were allowed to sit for a period of time, the samples remained in suspension. When the standard coal slurries were allowed to sit for a similar period of time, the solids settled and the water came to the top . It was as if the hydrolyzate created an emulsion with the coal and prevented the settling. This phenomenon is illustrated in Figure 38. Even thought the viscosity would be high, the advantage would be a mixture that resists settling.

The following figures illustrate the grinding performance of the coal slurries with respect to the particle size distribution. Figures 50, 51, 52, 53, and 54 illustrate the coal slurry performance for the temperature ranges of 170ºC, 160ºC, 150ºC, 140ºC respectively for sodium hydroxide lysing agent, and 160ºC for sulfuric acid lysing agent compared to the standard coal grind.

The temperature ranges that resulted in poor coal grinds were the 170°C sodium hydroxide samples, the 140ºC sodium hydroxide samples, and the 160ºC sulfuric acid samples. The main indicator for the poor results is that the tests had a high solids content in the lower mesh sizes and a low solids content in the high mesh sizes. Additionally, the set of tests had relatively high deviations. The tendency toward foaming let to poor performance in the 140ºC sodium hydroxide and the sulfuric acid grinds. The

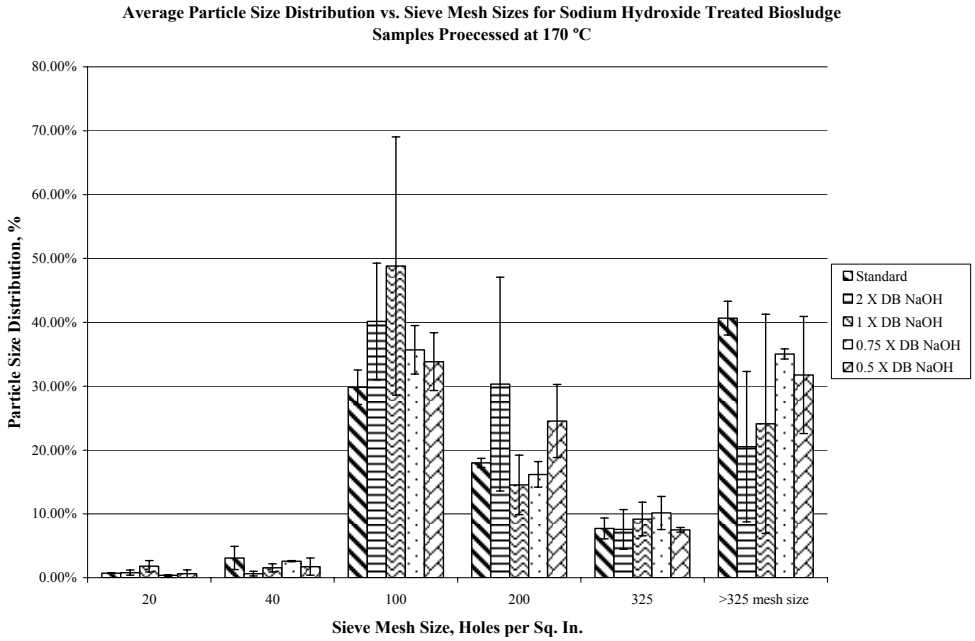


FIGURE 50 Average Particle Size Distribution with Respect to Sieve Mesh Sizes for Sodium Hydroxide Treated Biosludge Samples Processed at 170°C

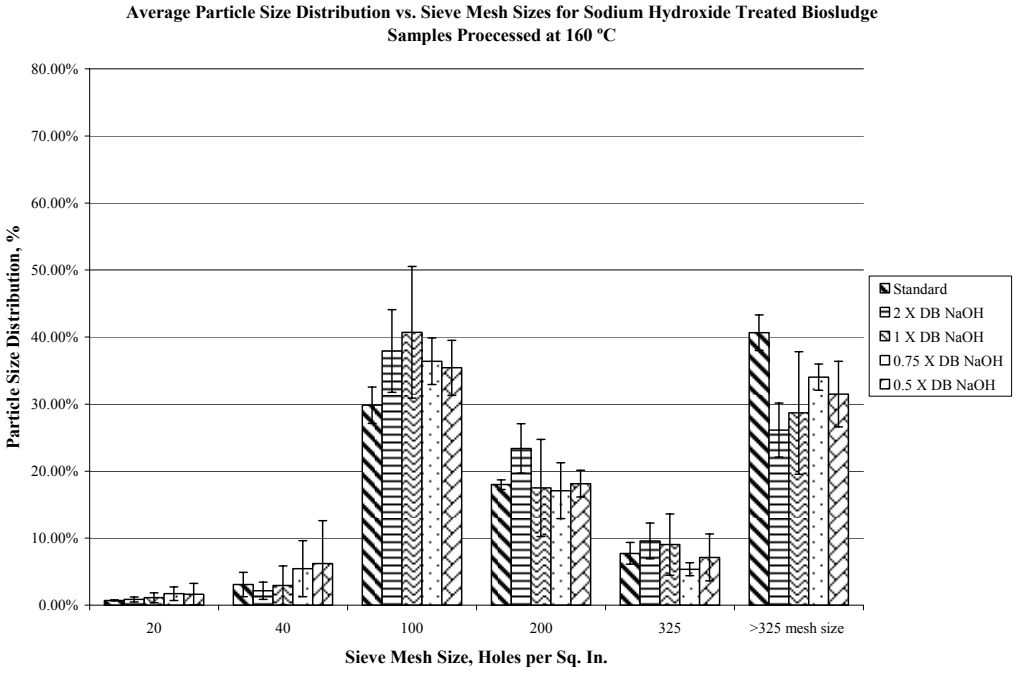


FIGURE 51 Average Particle Size Distribution with Respect to Sieve Mesh Sizes for Sodium Hydroxide Treated Biosludge Samples Processed at 160°C

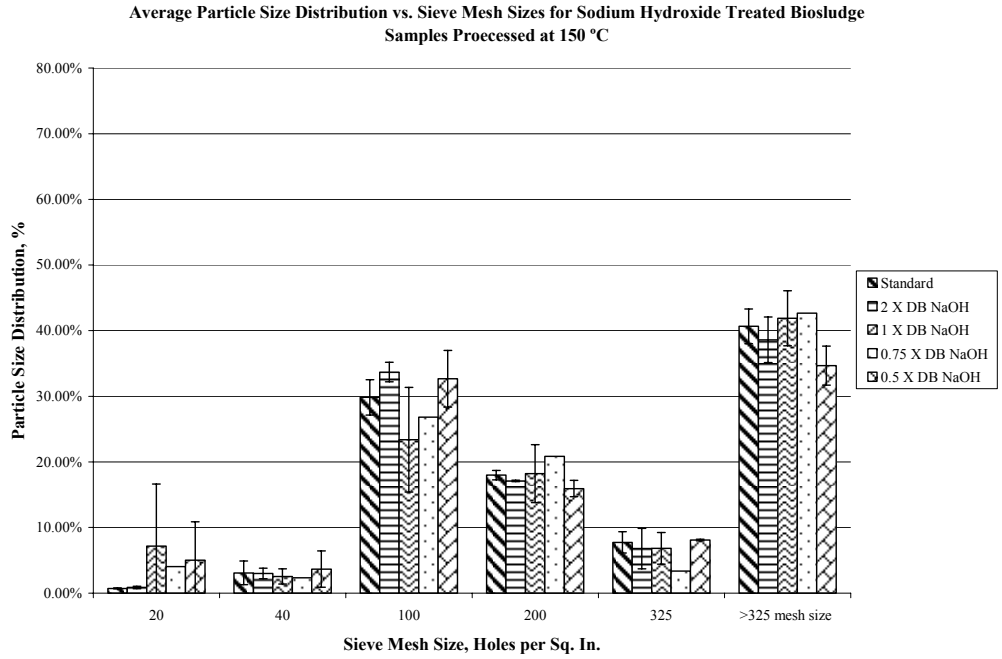


FIGURE 52 Average Particle Size Distribution with Respect to Sieve Mesh Sizes for Sodium Hydroxide Treated Biosludge Samples Processed at 150°C

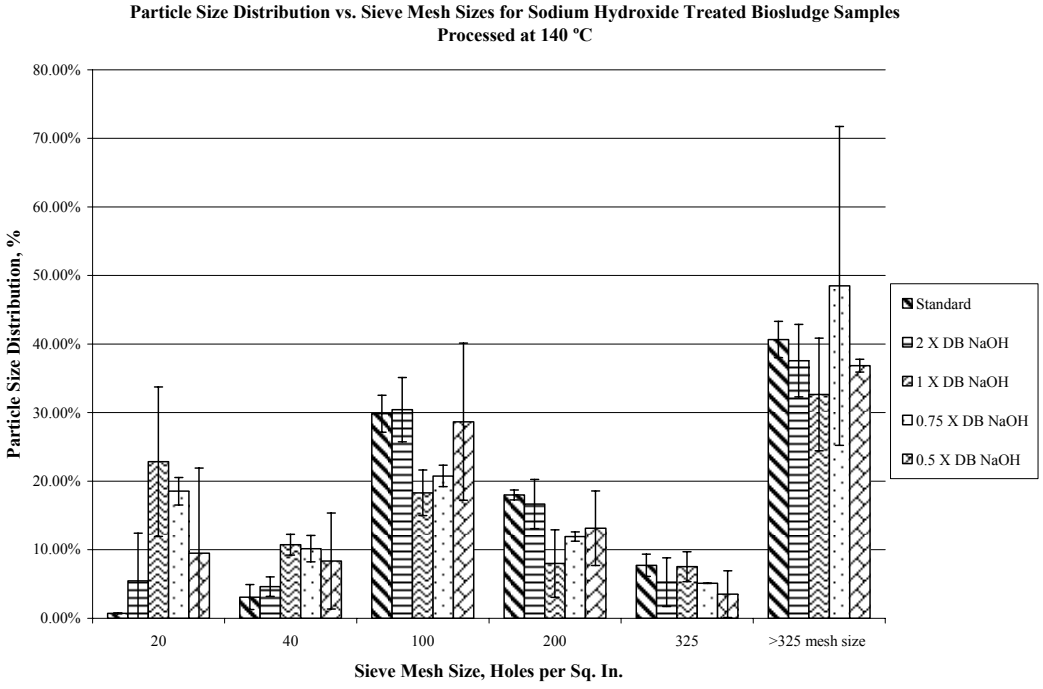


FIGURE 53 Average Particle Size Distribution with Respect to Sieve Mesh Sizes for Sodium Hydroxide Treated Biosludge Samples Processed at 140°C

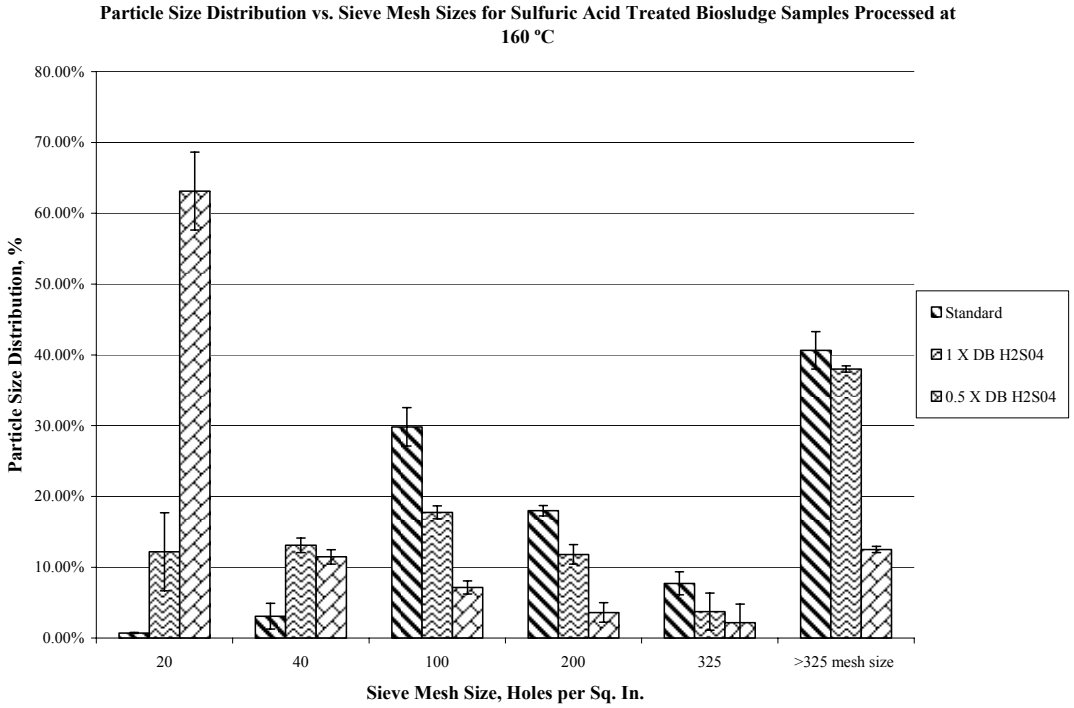


FIGURE 54 Average Particle Size Distribution with Respect to Sieve Mesh Sizes for Sulfuric Acid Treated Biosludge Samples Processed at 160°C

170ºC sodium hydroxide samples were very viscous. The samples at 160ºC temperature range had a slightly higher percentage of coal solids at the 100 mesh size and a lower percentage at solids greater than 325 mesh size. The samples at 150°C performed remarkably well with low deviation.

4.7 CONCLUSIONS

The total solids of the coal grinds were within the vicinity of 68% with the exception of a few samples with the sodium hydroxide, and the entire sample set for the sulfuric acid. The pH for the standard coal slurry was approximately 2.7. the sodium hydroxide slurry samples were greater than a pH of 10. The sulfuric acid slurry samples were less than a pH of 2. The average viscosity of standard coal slurry was approximately

4,000 cP. The low temperature range and high temperature range set of slurry samples had an average viscosity greater than 40,000 cP. The sulfuric acid low concentration had a viscosity greater than 100,000 cP. The sulfuric acid at 100% had a viscosity greater than 12,000 cP. The best average viscosity range for samples in the 150ºC and 160ºC samples was between 8,000 and 12,000 cP. The low and high temperature slurries of the sodium hydroxide hydrolyzate had poor grinding performance with respect to the standard coal slurry due to foaming and high viscosity, respectively. The sulfuric acid slurry samples were foamy and as a result had poor performance. The 160ºC sodium hydroxide slurry samples were slightly off specification. The 150º C sodium hydroxide slurry samples had optimal performance.

Overall, the 150ºC sodium hydroxide samples had an optimal balance of the properties best performance closely comparable to the performance of a standard coal slurry. The 160ºC samples had less than optimal performance. And, the remainder samples had very poor performance properties.

CHAPTER 5 SUMMARY OF CONCLUSIONS

5.1 OPERATION OF UNIT BX SUMMARY OF CONCLUSIONS

- 15 of the 18 experiments performed were within 5ºC on average with the selected experimental temperature.
- The longest duration of operation for the system over the summer of 2004 was 62 hours.
- 44 two quart samples of sodium hydroxide lysing agent samples were collected.
- 5 two quart samples of sulfuric acid lysing agent samples were collected.
- The average residence time of the system was 9.9 minutes.
- The variables that affected overall system performance are:
    - Biosludge Flow Rate
    - Lysing Agent Flow Rate
    - Liquid Level
- Primary sources of system upset and instability were:
    - Bridging of the feed biosludge
    - Erratic fluid flow control
    - Non-steady state heating
- Primary strategies for maximizing operational performance were:
    - Preventing bridging in the feed hopper
    - Using a Blow-Case valve arrangement to manage the non-Newtonian fluid properties of the treated sludge
    - Steam jacketing the process lines for heating up the biosludge prior to the reactor
- The primary variable that can shut down the system is the reactor pressure.
- The hydrolyzed biosludge is nutrient rich material that can be consumed by biological systems.
- It is possible to hydrolyze 15 weight percent solids biosludge in a continuous manner and create a water-like substance that is nutrient-rich.

5.2 EVALUATION OF A WASTEWATER TREATMENT SYSTEM WITH THE RECYCLE OF TREATED BIOSLUDGE SUMMARY OF CONCLUSIONS

- The best lysing agent and concentration for this option is 50% design basis sulfuric acid.
- The worst lysing agent and concentration is 200% sodium hydroxide.
- The optimal design basis for both lysing agents is 50%.

- The best scenario for recycling biosludge to WWT is to pre-filter the influent stream to remove solids.

5.3 SLURRYING HYDROLYZATE WITH COAL SUMMARY OF CONCLUSIONS

- The total solids of the coal grinds were on average 68% with the exception of a few samples.
- The pH of the standard coal slurries were approximately 2.7.
- The pH of the sodium hydroxide hydrolyzate slurries were greater than 10.
- The pH of the sulfuric acid hydrolyzate slurries were less the 2.
- The average viscosity of the standard coal slurry was approximately 4,000 cP .
- The low and high temperature sodium hydroxide hydrolyzate coal slurry samples had a viscosity greater than 40,000 cP.
- The sulfuric acid hydrolyzate coal slurries had a viscosity greater than 100,000 cP.
- The 150ºC and 160 ºC sodium hydroxide hydrolyzate slurries had a viscosity range between 8,000 and 12,000 cP.
- The low and high temperature sodium hydroxide hydrolyzate coal slurries and sulfuric acid hydrolyzate coal slurries had the worst particle size distribution compared to the coal slurry standards.
- The 160ºC samples particle size distribution were slightly off specification compared to the standard coal slurry particle size distribution.
- The 150ºC samples particle size distribution closely matched the performance of standard coal slurry.
- Overall, the 150ºC samples performed closely comparable to the standard coal slurry

CHAPTER 6 RECOMMENDATIONS

6.1 OPERATION OF UNIT BX SUMMARY OF RECOMMENDATIONS

- Assess the need to incorporate a controlled pressure relief system to reduce vapor space pressurization and release trapped gases.
- Investigate feed designs that will eliminate possible bridging and minimize strenuous labor activities to remove bridging.
- Develop a rigorous fluid model of the treated product for more effective fluid flow control.
- Develop a model of blow-case valve arrangement and determine the optimal sizing parameters for scalability purposes.
- Determine the key factors that affect product quality, uniformity, and reproducibility.
- Assess the impacts of the microorganism morphology on the system performance.
- Assess the impacts of different solids content of untreated sludge on system performance.
- Refine the process control for best practices of handling and processing sludge.
- Develop a better understanding of the rheological properties of high solids content sludge.
- Process sludge with inert plastic material that is normally present in WWT

6.2 EVALUATION OF A WASTEWATER TREATMENT SYSTEM WITH THE RECYCLE OF TREATED BIOSLUDGE SUMMARY OF RECOMMENDATIONS

- Assess the performance of the WWT with recycle with other possible lysing agents such as calcium hydroxide.
- Assess possible filtering methods of inerts WWT influent to accommodate recycle of treated hydrolyzate.
- Determine a clear relationship between the lysing agent concentration, treated hydrolyzate processing temperature, and the BOD_5 and TOC.

6.3 SLURRYING HYDROLYZATE WITH COAL SUMMARY OF RECOMMENDATIONS

- Develop a model that shows why the 150ºC sodium hydroxide hydrolyzate samples performed better than other samples.
- Develop a model of the low temperature and high temperature sodium hydroxide hydrolyzate slurries to show why these slurries did not perform well.

- Determine the reasons why the low temperature sodium hydroxide samples and sulfuric acid samples foam.
- Experiment further with the 150ºC sodium hydroxide hydrolyzate, varying concentrations of water and hydrolyzate.
- Test whether the concentration of solids from the hydrolyzate can be neglected while maintaining a 68% coal in the slurry.
- Determine the effects of neutralizing the sulfuric acid hydrolyzate prior to grinding with coal.
- Determine properties of the treated hydrolyzate that explain the suspension characteristics and emulsifying properties with coal and water.
- Develop methods to quantify the emulsifying properties of the hydrolyzate and coal slurry mixtures.

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WORKS CITED

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APPENDIX A UNIT BX SAMPLE PROCESS AND TEST DATA

APPENDIX A LIST OF SUPPLEMENTRY TABLES

TABLE 1 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 170 ºC 2 Hour Sample

Sample ID:	072104-JH-01	Sample Date:	7/21/2004
Sample Start Time:	15:40	Sample Finish Time:	15:50
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	170ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	167.31	±	0.1790	^{o}C
Reactor Pressure:	81.61	±	0.3366	psig
Flow Rate of Sludge:	545.06	±	2.6953	$gmin^{-1}$
Flow Rate of Caustic:	114.89	±	42.9601	$gmin^{-1}$
Hot Oil Supply Temperature:	195.78	±	0.1197	^{o}C
Hot Oil Return Temperature:	195.77	±	0.1616	^{o}C
Top Flange Temperature:	190.02	±	0.0015	^{o}C
Bottom Flange Temperature:	190.84	±	0.0225	^{o}C
Reactor Level:	4802.83	±	376.1490	mL
Residence Time:	7.32	±	0.8369	min
Caustic Sludge Ratio:	0.2123	±	0.0200	unitless
Percent Solids				
Sample 1 wt%			28.52	%
Sample 1 wt%			28.66	%
Sample 1 wt%			28.72	%
Average			28.63	%
Stand. Dev.			0.1026	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	9.3	9300
0.6	12.2	6100
1.5	14.5	2900
3	18.1	1810
6	21.8	1090
12	26.6	665
30	35	350
60	43	215
Spindle #	2	

Reference coal grinds from pages 84-7, 94-6 in notebook 1

TABLE 2 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 4 Hour Sample

Sample ID:	072104-JH-02	Sample Date:	7/21/2004
Sample Start Time:	17:40	Sample Finish Time:	17:50
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	170ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	168.53	±	0.0725	°C
Reactor Pressure:	99.60	±	0.9536	psig
Flow Rate of Sludge:	548.75	±	2.0663	$gmin^{-1}$
Flow Rate of Caustic:	114.04	±	5.6687	$gmin^{-1}$
Hot Oil Supply Temperature:	211.62	±	0.0152	°C
Hot Oil Return Temperature:	211.59	±	0.0248	°C
Top Flange Temperature:	210.35	±	0.0037	°C
Bottom Flange Temperature:	209.68	±	0.0014	°C
Reactor Level:	4953.44	±	152.4930	mL
Residence Time:	7.48	±	0.2464	min
Caustic Sludge Ratio:	0.2078	±	0.0035	unitless

Percent Solids		
Sample 1 wt%	22.64	%
Sample 1 wt%	22.6	%
Sample 1 wt%	22.6	%
Average	22.61	%
Stand. Dev.	0.0231	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	21.1	4220
0.6	24.6	2460
1.5	30.6	1224
3	36	720
6	43	430
12	52.1	260.5
30	70.5	141
60	96.4	96.4
Spindle #	1	

Reference coal grinds from pages 89-93 in notebook 1

TABLE 3 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 6 Hour Sample

Sample ID:	072104-JH-03	Sample Date:	7/21/2004
Sample Start Time:	19:40	Sample Finish Time:	19:50
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	170ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	167.23	±	0.2032	°C
Reactor Pressure:	89.92	±	0.2678	psig
Flow Rate of Sludge:	550.35	±	1.8951	$gmin^{-1}$
Flow Rate of Caustic:	114.08	±	1.8480	$gmin^{-1}$
Hot Oil Supply Temperature:	212.25	±	0.0151	°C
Hot Oil Return Temperature:	212.61	±	0.0247	°C
Top Flange Temperature:	210.19	±	0.0037	°C
Bottom Flange Temperature:	209.63	±	0.0014	°C
Reactor Level:	4749.69	±	88.9196	mL
Residence Time:	7.15	±	0.1385	min
Caustic Sludge Ratio:	0.2073	±	0.0034	unitless

Percent Solids		
Sample 1 wt%	24.584	%
Sample 1 wt%	23.45	%
Sample 1 wt%	23.316	%
Average	23.78	%
Stand. Dev.	0.6966	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	22.6	4520
0.6	27.4	2740
1.5	34.9	1396
3	42	840
6	49.2	492
12	58.3	291.5
30	76.7	153.4
60	---	---
Spindle #	1	

Reference coal grinds from pages 97-100 in notebook 1

TABLE 4 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 8 Hour Sample

Sample ID:	072104-JH-04	Sample Date:	7/21/2004
Sample Start Time:	21:40	Sample Finish Time:	21:50
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	170ºC	Sample Time:	8 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	164.49	±	0.1617	°C
Reactor Pressure:	86.38	±	0.5641	psig
Flow Rate of Sludge:	550.78	±	1.6805	$gmin^{-1}$
Flow Rate of Caustic:	114.25	±	9.5516	$gmin^{-1}$
Hot Oil Supply Temperature:	216.69	±	0.0046	°C
Hot Oil Return Temperature:	217.28	±	0.0031	°C
Top Flange Temperature:	210.04	±	0.0037	°C
Bottom Flange Temperature:	209.57	±	0.0014	°C
Reactor Level:	5533.01	±	89.5254	mL
Residence Time:	8.32	±	0.2009	min
Caustic Sludge Ratio:	0.2075	±	0.0039	unitless

Percent Solids		
Sample 1 wt%	22.38	%
Sample 1 wt%	21.7	%
Sample 1 wt%	22.1	%
Average	22.06	%
Stand. Dev.	0.3418	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	22.9	4580
0.6	27.9	2790
1.5	35.1	1404
3	43	860
6	50.7	507
12	60.8	304
30	80.6	161.2
60	---	---
Spindle #	1	

Reference coal grinds from pages 103-106 in notebook 1

TABLE 5 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 2 Hour Sample

Sample ID:	072204-NB-01	Sample Date:	7/22/2004
Sample Start Time:	15:00	Sample Finish Time:	15:10
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	170ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	172.50	±	0.0391	°C
Reactor Pressure:	111.08	±	0.4501	psig
Flow Rate of Sludge:	549.58	±	1.7596	$gmin^{-1}$
Flow Rate of Caustic:	58.07	±	1.6962	$gmin^{-1}$
Hot Oil Supply Temperature:	221.12	±	0.0586	°C
Hot Oil Return Temperature:	221.83	±	0.3013	°C
Top Flange Temperature:	215.24	±	0.0362	°C
Bottom Flange Temperature:	215.19	±	0.0007	°C
Reactor Level:	4521.95	±	276.2233	mL
Residence Time:	7.44	±	0.4638	min
Caustic Sludge Ratio:	0.1057	±	0.0032	unitless

Percent Solids		
Sample 1 wt%	25.59	%
Sample 1 wt%	25.1	%
Sample 1 wt%	22.23	%
Average	24.31	%
Stand. Dev.	1.8151	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	7.5	150
0.6	11.5	115
1.5	18.2	72.8
3	27.4	54.8
6	40.1	40.1
12	60	30
30	---	---
60	---	---
Spindle #	UL	

Reference coal grinds from pages 109-12 in notebook 1

TABLE 6 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 4 Hour Sample

Sample ID:	072204-NB-02	Sample Date:	7/22/2004
Sample Start Time:	17:00	Sample Finish Time:	17:10
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	170ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	172.40	±	0.0156	°C
Reactor Pressure:	106.34	±	0.4688	psig
Flow Rate of Sludge:	549.70	±	2.1743	$gmin^{-1}$
Flow Rate of Caustic:	57.28	±	1.4591	$gmin^{-1}$
Hot Oil Supply Temperature:	222.29	±	0.0000	°C
Hot Oil Return Temperature:	222.70	±	0.0010	°C
Top Flange Temperature:	214.98	±	0.0005	°C
Bottom Flange Temperature:	215.16	±	0.0007	°C
Reactor Level:	4635.30	±	92.8103	mL
Residence Time:	7.64	±	0.1447	min
Caustic Sludge Ratio:	0.1042	±	0.0040	unitless

Percent Solids		
Sample 1 wt%		%
Sample 1 wt%	29.87	%
Sample 1 wt%	30.53	%
Average	30.20	%
Stand. Dev.	0.4667	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	9.9	198
0.6	15.4	154
1.5	24.1	96.4
3	34	68
6	49.8	49.8
12	74.2	37.1
30	---	---
60	---	---
Spindle #	UL	

Reference coal grinds from pages 115-18 in notebook 1

TABLE 7 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 6 Hour Sample

Sample ID:	072204-NB-03	Sample Date:	7/22/2004
Sample Start Time:	19:00	Sample Finish Time:	19:10
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	170ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	168.11	±	0.0292	°C
Reactor Pressure:	103.63	±	0.3126	psig
Flow Rate of Sludge:	550.41	±	2.2360	$gmin^{-1}$
Flow Rate of Caustic:	57.02	±	1.3554	$gmin^{-1}$
Hot Oil Supply Temperature:	222.29	±	0.0000	°C
Hot Oil Return Temperature:	222.66	±	0.0010	°C
Top Flange Temperature:	215.00	±	0.0005	°C
Bottom Flange Temperature:	215.13	±	0.0007	°C
Reactor Level:	5708.88	±	24.6605	mL
Residence Time:	9.40	±	0.0641	min
Caustic Sludge Ratio:	0.1036	±	0.0040	unitless

Percent Solids		
Sample 1 wt%	14.45	%
Sample 1 wt%	13.65	%
Sample 1 wt%	13.47	%
Average	13.86	%
Stand. Dev.	0.5217	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
Spindle #	N/A	

Reference coal grinds from pages 121-24 in notebook 1

TABLE 8 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 8 Hour Sample

Sample ID:	072204-JH-04	Sample Date:	7/22/2004
Sample Start Time:	21:00	Sample Finish Time:	21:10
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	170ºC	Sample Time:	8 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	166.01	±	0.1224	°C
Reactor Pressure:	109.01	±	3.9553	psig
Flow Rate of Sludge:	549.65	±	1.3829	$gmin^{-1}$
Flow Rate of Caustic:	57.10	±	15.8338	$gmin^{-1}$
Hot Oil Supply Temperature:	222.29	±	0.0000	°C
Hot Oil Return Temperature:	222.62	±	0.0010	°C
Top Flange Temperature:	215.02	±	0.0005	°C
Bottom Flange Temperature:	215.06	±	0.0150	°C
Reactor Level:	5698.71	±	220.6632	mL
Residence Time:	9.40	±	0.3738	min
Caustic Sludge Ratio:	0.1040	±	0.0043	unitless
Percent Solids				
Sample 1 wt%			14.37	%
Sample 1 wt%			14.38	%
Sample 1 wt%			14.29	%
Average			14.35	%
Stand. Dev.			0.0493	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	15.3	306
0.6	22.2	222
1.5	34.8	139.2
3	51.6	103.2
6	70.2	70.2
12	---	---
30	---	---
60	---	---
Spindle #	UL	

Reference coal grinds from pages 127-130 in notebook 1

TABLE 9 System Data and Analytical Measurements with 75% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 2 Hour Sample

Sample ID:	072304-JH-01	Sample Date:		7/23/2004
Sample Start Time:	1:19	Sample Finish Time:		1:29
Lysing Agent:	NaOH	Design Basis:		75%
Experiment Temp:	170ºC	Sample Time:		2 hours
System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	161.10	±	0.0949	°C
Reactor Pressure:	102.35	±	1.2406	psig
Flow Rate of Sludge:	525.94	±	1.6963	$gmin^{-1}$
Flow Rate of Caustic:	41.19	±	1.9936	$gmin^{-1}$
Hot Oil Supply Temperature:	222.12	±	0.0061	°C
Hot Oil Return Temperature:	222.44	±	0.0031	°C
Top Flange Temperature:	215.02	±	0.0005	°C
Bottom Flange Temperature:	225.61	±	0.0028	°C
Reactor Level:	5901.60	±	18.8189	mL
Residence Time:	10.41	±	0.0605	min
Caustic Sludge Ratio:	0.0783	±	0.0031	unitless
Percent Solids				
Sample 1 wt%			4.266	%
Sample 1 wt%			6.57	%
Sample 1 wt%			4.283	%
Average			5.04	%
Stand. Dev.			1.3253	%
Viscosity Measurements				
RPM, s^{-1}	Percent Torque, %		Viscosity, cP	
0.3	25		5000	
0.6	32.6		3260	
1.5	42.1		1684	
3	52.7		1054	
6	68.1		681	
12	88.6		443	
30	---		---	
60	---		---	
Spindle #	1			

Reference coal grinds from pages 133-136 in notebook 1

TABLE 10 System Data and Analytical Measurements with 75% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 4 Hour Sample

Sample ID:	072304-JH-02	Sample Date:	7/23/2004
Sample Start Time:	3:40	Sample Finish Time:	3:50
Lysing Agent:	NaOH	Design Basis:	75%
Experiment Temp:	170ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	173.56	±	0.5669	°C
Reactor Pressure:	116.22	±	1.5011	psig
Flow Rate of Sludge:	514.61	±	1.8982	$gmin^{-1}$
Flow Rate of Caustic:	40.26	±	18.4571	$gmin^{-1}$
Hot Oil Supply Temperature:	221.83	±	0.0061	°C
Hot Oil Return Temperature:	222.29	±	0.0031	°C
Top Flange Temperature:	215.00	±	0.0005	°C
Bottom Flange Temperature:	225.75	±	0.0028	°C
Reactor Level:	5631.17	±	88.2911	mL
Residence Time:	10.16	±	0.4279	min
Caustic Sludge Ratio:	0.0783	±	0.0039	unitless

Percent Solids		
Sample 1 wt%	8.949	%
Sample 1 wt%	9.44	%
Sample 1 wt%	8.861	%
Average	9.08	%
Stand. Dev.	0.3120	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	6.9	1380
0.6	10.6	1060
1.5	14.3	572
3	19.4	388
6	25.4	254
12	33.6	168
30	48.5	97
60	62	62
Spindle #	1	

Reference coal grinds from pages 139-42 in notebook 1

TABLE 11 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 2 Hour Sample

Sample ID:	070804-NB-01	Sample Date:	7/8/2004
Sample Start Time:	15:40	Sample Finish Time:	15:50
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	170ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	172.18	±	0.1547	°C
Reactor Pressure:	105.31	±	0.1102	psig
Flow Rate of Sludge:	395.92	±	1.3159	$gmin^{-1}$
Flow Rate of Caustic:	23.45	±	7.1142	$gmin^{-1}$
Hot Oil Supply Temperature:	184.84	±	0.0065	°C
Hot Oil Return Temperature:	185.41	±	0.0000	°C
Top Flange Temperature:	188.74	±	0.0055	°C
Bottom Flange Temperature:	189.51	±	0.0023	°C
Reactor Level:	4078.49	±	68.5877	mL
Residence Time:	9.73	±	0.2214	min
Caustic Sludge Ratio:	0.0592	±	0.0034	unitless

Percent Solids		
Sample 1 wt%	12.54	%
Sample 1 wt%	12.58	%
Sample 1 wt%	12.11	%
Average	12.41	%
Stand. Dev.	0.2606	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	2.1	42
0.6	3.5	35
1.5	5.8	23.2
3	9.1	18.2
6	13.1	13.1
12	18.3	9.15
30	36.2	7.22
60	63.6	6.32
Spindle #	UL	

Reference coal grinds from pages 151-54 in notebook 1

TABLE 12 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 170°C 4 Hour Sample

Sample ID:	070804-NB-02	Sample Date:	7/8/2004
Sample Start Time:	16:40	Sample Finish Time:	16:50
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	170ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	167.91	±	0.3348	°C
Reactor Pressure:	94.06	±	0.9644	psig
Flow Rate of Sludge:	398.56	±	1.2590	$gmin^{-1}$
Flow Rate of Caustic:	23.74	±	2.1238	$gmin^{-1}$
Hot Oil Supply Temperature:	184.98	±	0.0065	°C
Hot Oil Return Temperature:	185.41	±	0.0000	°C
Top Flange Temperature:	188.63	±	0.0055	°C
Bottom Flange Temperature:	189.47	±	0.0023	°C
Reactor Level:	4346.34	±	42.2490	mL
Residence Time:	10.29	±	0.0983	min
Caustic Sludge Ratio:	0.0596	±	0.0032	unitless

Percent Solids		
Sample 1 wt%	12.61	%
Sample 1 wt%	12.56	%
Sample 1 wt%	12.43	%
Average	12.53	%
Stand. Dev.	0.0929	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	1.1	22
0.6	1.8	18
1.5	2.6	10.4
3	3.2	6.4
6	4.4	4.4
12	7.6	3.8
30	16.5	3.28
60	31.5	3.11
Spindle #	UL	

Reference coal grinds from pages 155-58 in notebook 1

TABLE 13 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 2 Hour Sample

Sample ID:	080604-MV-08	Sample Date:	8/6/2004
Sample Start Time:	18:30	Sample Finish Time:	18:40
Lysing Agent:	NaOH	Design Basis:	200
Experiment Temp:	160ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	164.49	±	0.1676	°C
Reactor Pressure:	116.14	±	2.0396	psig
Flow Rate of Sludge:	426.72	±	1.5961	$gmin^{-1}$
Flow Rate of Caustic:	89.98	±	10.4736	$gmin^{-1}$
Hot Oil Supply Temperature:	235.20	±	0.0106	°C
Hot Oil Return Temperature:	235.72	±	0.0183	°C
Top Flange Temperature:	225.00	±	0.0005	°C
Bottom Flange Temperature:	229.58	±	0.0142	°C
Reactor Level:	4428.54	±	79.1360	mL
Residence Time:	8.58	±	0.2418	min
Caustic Sludge Ratio:	0.2110	±	0.0051	unitless

Percent Solids		
Sample 1 wt%	19.16	%
Sample 1 wt%	19	%
Sample 1 wt%	19.47	%
Average	19.21	%
Stand. Dev.	0.2390	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	2.5	2500
0.6	3.5	1750
1.5	4.6	920
3	5.9	590
6	7.8	390
12	10.9	272.5
30	17	170
60	23.7	118.5
Spindle #	2	

Reference coal grinds from pages 159-62 in notebook 1

TABLE 14 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 4 Hour Sample

Sample ID:	080604-JH-09	Sample Date:	8/6/2004
Sample Start Time:	20:30	Sample Finish Time:	20:40
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	160ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	157.63	±	0.0187	°C
Reactor Pressure:	115.96	±	3.7368	psig
Flow Rate of Sludge:	411.79	±	3.2814	$gmin^{-1}$
Flow Rate of Caustic:	86.48	±	21.7144	$gmin^{-1}$
Hot Oil Supply Temperature:	236.63	±	0.0789	°C
Hot Oil Return Temperature:	237.32	±	0.0804	°C
Top Flange Temperature:	224.98	±	0.0005	°C
Bottom Flange Temperature:	224.95	±	0.1099	°C
Reactor Level:	4770.02	±	84.8805	mL
Residence Time:	9.60	±	0.5167	min
Caustic Sludge Ratio:	0.2104	±	0.0106	unitless

Percent Solids		
Sample 1 wt%	18.7	%
Sample 1 wt%	18.9	%
Sample 1 wt%	18.98	%
Average	18.86	%
Stand. Dev.	0.1442	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	18.7	3740
0.6	24.5	2450
1.5	32	1280
3	39.6	792
6	49.9	499
12	68.5	342.5
30	---	---
60	---	---
Spindle #	1	

Reference coal grinds from pages 163-66 in notebook 1

TABLE 15 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 6 Hour Sample

Sample ID:	080604-JH-10	Sample Date:	8/6/2004
Sample Start Time:	22:30	Sample Finish Time:	22:40
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	160ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	173.31	±	1.1792	°C
Reactor Pressure:	144.99	±	4.0886	psig
Flow Rate of Sludge:	452.62	±	18.9282	$gmin^{-1}$
Flow Rate of Caustic:	83.51	±	152.3495	$gmin^{-1}$
Hot Oil Supply Temperature:	240.64	±	0.1608	°C
Hot Oil Return Temperature:	241.48	±	0.2770	°C
Top Flange Temperature:	224.96	±	0.0005	°C
Bottom Flange Temperature:	224.86	±	0.0005	°C
Reactor Level:	3429.40	±	449.1732	mL
Residence Time:	6.92	±	2.1492	min
Caustic Sludge Ratio:	0.1972	±	0.0610	unitless

Percent Solids		
Sample 1 wt%	21.26	%
Sample 1 wt%	21.56	%
Sample 1 wt%	21.65	%
Average	21.49	%
Stand. Dev.	0.2042	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	25.2	5040
0.6	31.4	3140
1.5	37.5	1500
3	43.9	878
6	54.4	544
12	70.1	350.5
30	---	---
60	---	---
Spindle #	1	

Reference coal grinds from pages 167-70 in notebook 1

TABLE 16 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 8 Hour Sample

Sample ID:	080704-JH-01	Sample Date:	8/7/2004
Sample Start Time:	0:30	Sample Finish Time:	0:40
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	160ºC	Sample Time:	8 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	149.95	±	0.1608	°C
Reactor Pressure:	130.19	±	2.7365	psig
Flow Rate of Sludge:	447.68	±	1.8266	$gmin^{-1}$
Flow Rate of Caustic:	94.83	±	13.7189	$gmin^{-1}$
Hot Oil Supply Temperature:	221.20	±	0.0098	°C
Hot Oil Return Temperature:	221.76	±	0.0170	°C
Top Flange Temperature:	224.94	±	0.0005	°C
Bottom Flange Temperature:	224.89	±	0.0005	°C
Reactor Level:	4509.86	±	73.4938	mL
Residence Time:	8.32	±	0.2639	min
Caustic Sludge Ratio:	0.2119	±	0.0052	unitless

Percent Solids		
Sample 1 wt%	19.64	%
Sample 1 wt%	18.96	%
Sample 1 wt%	19.14	%
Average	19.25	%
Stand. Dev.	0.3523	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	11.6	2320
0.6	14	1400
1.5	19.7	788
3	27.4	548
6	36.6	366
12	48.8	244
30	72.4	144.8
60	99.9	99.9
Spindle #	1	

Reference coal grinds from pages 171-74 in notebook 1

TABLE 17 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 2 Hour Sample

Sample ID:	080604-NB-05	Sample Date:	8/6/2004
Sample Start Time:	10:50	Sample Finish Time:	11:00
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	160ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	162.79	±	0.0554	°C
Reactor Pressure:	104.23	±	1.3097	psig
Flow Rate of Sludge:	494.64	±	1.7854	$gmin^{-1}$
Flow Rate of Caustic:	52.45	±	7.9229	$gmin^{-1}$
Hot Oil Supply Temperature:	223.87	±	0.0000	°C
Hot Oil Return Temperature:	224.40	±	0.0000	°C
Top Flange Temperature:	224.86	±	0.0010	°C
Bottom Flange Temperature:	224.90	±	0.0015	°C
Reactor Level:	4460.85	±	72.1774	mL
Residence Time:	8.16	±	0.1683	min
Caustic Sludge Ratio:	0.1061	±	0.0039	unitless

Percent Solids		
Sample 1 wt%	16.08	%
Sample 1 wt%	15.51	%
Sample 1 wt%	15.45	%
Average	15.68	%
Stand. Dev.	0.3477	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	11.1	2220
0.6	15.3	1530
1.5	21.3	852
3	27.5	550
6	35.8	358
12	47.5	237.5
30	69.2	138.4
60	92.5	92.5
Spindle #	1	

Reference coal grinds from pages 175-78 in notebook 1

TABLE 18 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 4 Hour Sample

Sample ID:	080604-NB-06	Sample Date:	8/6/2004
Sample Start Time:	14:15	Sample Finish Time:	14:25
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	160ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	162.79	±	0.0554	°C
Reactor Pressure:	104.23	±	1.3097	psig
Flow Rate of Sludge:	494.64	±	1.7854	$gmin^{-1}$
Flow Rate of Caustic:	52.45	±	7.9229	$gmin^{-1}$
Hot Oil Supply Temperature:	223.87	±	0.0000	°C
Hot Oil Return Temperature:	224.40	±	0.0000	°C
Top Flange Temperature:	224.86	±	0.0010	°C
Bottom Flange Temperature:	224.90	±	0.0015	°C
Reactor Level:	4460.85	±	72.1774	mL
Residence Time:	8.16	±	0.1683	min
Caustic Sludge Ratio:	0.1061	±	0.0039	unitless

Percent Solids		
Sample 1 wt%	13.33	%
Sample 1 wt%	13.33	%
Sample 1 wt%	12.69	%
Average	13.12	%
Stand. Dev.	0.3695	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	7	140
0.6	12.7	127
1.5	16.7	66.8
3	21.7	43.4
6	36.2	36.2
12	56.3	28.15
30	98.1	19.6
60	---	---
Spindle #	UL	

Reference coal grinds from pages 179-82 in notebook 1

TABLE 19 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 6 Hour Sample

Sample ID:	080604-NB-07	Sample Date:	8/6/2004
Sample Start Time:	16:00	Sample Finish Time:	16:10
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	160ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	159.85	±	0.1392	°C
Reactor Pressure:	100.97	±	1.7499	psig
Flow Rate of Sludge:	393.79	±	1.6939	$gmin^{-1}$
Flow Rate of Caustic:	41.51	±	13.3964	$gmin^{-1}$
Hot Oil Supply Temperature:	225.56	±	0.1280	°C
Hot Oil Return Temperature:	226.21	±	0.1373	°C
Top Flange Temperature:	224.97	±	0.0010	°C
Bottom Flange Temperature:	228.84	±	0.0144	°C
Reactor Level:	4678.63	±	70.1109	mL
Residence Time:	10.76	±	0.4145	min
Caustic Sludge Ratio:	0.1055	±	0.0050	unitless

Percent Solids		
Sample 1 wt%	13.93	%
Sample 1 wt%	13.98	%
Sample 1 wt%	13.93	%
Average	13.95	%
Stand. Dev.	0.0289	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	10.4	2080
0.6	14.1	1410
1.5	18.8	752
3	25.1	502
6	33.4	334
12	45	225
30	66.1	132.2
60	88.7	88.7
Spindle #	1	

Reference coal grinds from pages 183-86 in notebook 1

TABLE 20 System Data and Analytical Measurements with 75% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 2 Hour Sample

Sample ID:	080604-JH-02	Sample Date:	8/6/2004
Sample Start Time:	4:45	Sample Finish Time:	4:55
Lysing Agent:	NaOH	Design Basis:	75%
Experiment Temp:	160ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	164.06	±	0.2401	°C
Reactor Pressure:	133.06	±	2.1710	psig
Flow Rate of Sludge:	470.36	±	3.0282	$gmin^{-1}$
Flow Rate of Caustic:	37.79	±	17.7933	$gmin^{-1}$
Hot Oil Supply Temperature:	218.40	±	0.0970	°C
Hot Oil Return Temperature:	218.76	±	0.1258	°C
Top Flange Temperature:	224.69	±	0.0015	°C
Bottom Flange Temperature:	224.88	±	0.0032	°C
Reactor Level:	4591.15	±	94.3091	mL
Residence Time:	9.05	±	0.4385	min
Caustic Sludge Ratio:	0.0805	±	0.0076	unitless
Percent Solids				
Sample 1 wt%			14.75	%
Sample 1 wt%			14.75	%
Sample 1 wt%			13.77	%
Average			14.42	%
Stand. Dev.			0.5658	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	13.3	2660
0.6	18.4	1840
1.5	25.2	1008
3	32.5	650
6	42.2	422
12	55.8	279
30	80.1	160.2
60	---	---
Spindle #	1	

Reference coal grinds from pages 187-90 in notebook 1

TABLE 21 System Data and Analytical Measurements with 75% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 4 Hour Sample

Sample ID:	080604-JH-03	Sample Date:	8/6/2004
Sample Start Time:	6:45	Sample Finish Time:	6:55
Lysing Agent:	NaOH	Design Basis:	75%
Experiment Temp:	160ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	160.99	±	0.0892	°C
Reactor Pressure:	116.81	±	2.0248	psig
Flow Rate of Sludge:	476.34	±	3.1338	$gmin^{-1}$
Flow Rate of Caustic:	38.02	±	17.8150	$gmin^{-1}$
Hot Oil Supply Temperature:	222.43	±	0.0440	°C
Hot Oil Return Temperature:	223.42	±	0.0244	°C
Top Flange Temperature:	224.75	±	0.0015	°C
Bottom Flange Temperature:	224.77	±	0.0015	°C
Reactor Level:	4751.20	±	66.0217	mL
Residence Time:	9.25	±	0.3317	min
Caustic Sludge Ratio:	0.0799	±	0.0075	unitless
Percent Solids				
Sample 1 wt%			13.07	%
Sample 1 wt%			13.17	%
Sample 1 wt%			13.09	%
Average			13.11	%
Stand. Dev.			0.0529	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	22.1	4420
0.6	29.7	2970
1.5	39	1560
3	48.7	974
6	63	630
12	81.7	408.5
30	---	---
60	---	---
Spindle #	1	

Reference coal grinds from pages 191-93 in notebook 1

TABLE 22 System Data and Analytical Measurements with 75% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 6 Hour Sample

Sample ID:	080604-JH-04	Sample Date:	8/6/2004
Sample Start Time:	8:50	Sample Finish Time:	9:00
Lysing Agent:	NaOH	Design Basis:	75%
Experiment Temp:	160ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	162.68	±	0.5166	°C
Reactor Pressure:	111.91	±	1.7682	psig
Flow Rate of Sludge:	534.14	±	5.7755	$gmin^{-1}$
Flow Rate of Caustic:	46.05	±	58.8259	$gmin^{-1}$
Hot Oil Supply Temperature:	224.12	±	0.0722	°C
Hot Oil Return Temperature:	224.51	±	0.1289	°C
Top Flange Temperature:	224.81	±	0.0014	°C
Bottom Flange Temperature:	224.84	±	0.0015	°C
Reactor Level:	4566.84	±	86.0410	mL
Residence Time:	7.95	±	0.7728	min
Caustic Sludge Ratio:	0.0868	±	0.0122	unitless
Percent Solids				
Sample 1 wt%	13.7			%
Sample 1 wt%	13.54			%
Sample 1 wt%	13.5			%
Average	13.58			%
Stand. Dev.	0.1058			%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	4.1	4100
0.6	6.2	3100
1.5	8.8	1760
3	11.1	1110
6	14.5	725
12	19.6	490
30	28.1	281
60	37.2	186
Spindle #	2	

Reference coal grinds from pages 195-97 in notebook 1

TABLE 23 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 2 Hour Sample

Sample ID:	080504-MV-06	Sample Date:	8/5/2004
Sample Start Time:	18:30	Sample Finish Time:	18:40
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	160º	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	158.47	±	0.0503	°C
Reactor Pressure:	80.73	±	0.4041	psig
Flow Rate of Sludge:	474.66	±	1.8484	$gmin^{-1}$
Flow Rate of Caustic:	24.32	±	1.0751	$gmin^{-1}$
Hot Oil Supply Temperature:	223.81	±	0.0007	°C
Hot Oil Return Temperature:	224.32	±	0.0000	°C
Top Flange Temperature:	224.44	±	0.0012	°C
Bottom Flange Temperature:	225.49	±	0.0025	°C
Reactor Level:	4883.98	±	53.2829	mL
Residence Time:	9.79	±	0.1021	min
Caustic Sludge Ratio:	0.0512	±	0.0039	unitless
Percent Solids				
Sample 1 wt%			13.29	%
Sample 1 wt%			13.25	%
Sample 1 wt%			13.32	%
Average			13.29	%
Stand. Dev.			0.0351	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	11.8	11800
0.6	18	9000
1.5	29.5	5900
3	36	3600
6	44.1	2205
12	56.1	1402.5
30	78.8	788
60	---	---
Spindle #	2	

Reference coal grinds from pages 200-06 in notebook 1

TABLE 24 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 4 Hour Sample

Sample ID:	080504-JH-07	Sample Date:	8/5/2004
Sample Start Time:	20:30	Sample Finish Time:	20:40
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	160ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	155.08	±	0.0100	°C
Reactor Pressure:	77.45	±	0.6999	psig
Flow Rate of Sludge:	475.05	±	3.1054	$gmin^{-1}$
Flow Rate of Caustic:	24.60	±	3.2865	$gmin^{-1}$
Hot Oil Supply Temperature:	223.84	±	0.0007	°C
Hot Oil Return Temperature:	224.32	±	0.0000	°C
Top Flange Temperature:	224.39	±	0.0012	°C
Bottom Flange Temperature:	225.34	±	0.0012	°C
Reactor Level:	5002.46	±	66.5127	mL
Residence Time:	10.01	±	0.1336	min
Caustic Sludge Ratio:	0.0518	±	0.0065	unitless

Percent Solids		
Sample 1 wt%	12.39	%
Sample 1 wt%	12.35	%
Sample 1 wt%	12.4	%
Average	12.38	%
Stand. Dev.	0.0265	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	10.8	10800
0.6	15.2	7600
1.5	20.6	4120
3	25.5	2550
6	33.1	1655
12	42.3	1057.5
30	62.5	625
60	83.1	415.5
Spindle #	2	

Reference coal grinds from pages 198-99 in notebook 1

TABLE 25 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 6 Hour Sample

Sample ID:	080504-JH-08	Sample Date:	8/5/2004
Sample Start Time:	22:20	Sample Finish Time:	22:30
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	160ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	164.46	±	3.1380	°C
Reactor Pressure:	94.32	±	7.4040	psig
Flow Rate of Sludge:	373.02	±	3.4676	$gmin^{-1}$
Flow Rate of Caustic:	20.02	±	98.8611	$gmin^{-1}$
Hot Oil Supply Temperature:	223.87	±	0.0007	°C
Hot Oil Return Temperature:	224.32	±	0.0000	°C
Top Flange Temperature:	224.35	±	0.0012	°C
Bottom Flange Temperature:	225.30	±	0.0012	°C
Reactor Level:	4657.92	±	249.3604	mL
Residence Time:	12.65	±	3.3344	min
Caustic Sludge Ratio:	0.0561	±	0.0116	unitless

Percent Solids		
Sample 1 wt%	12.9	%
Sample 1 wt%	13.66	%
Sample 1 wt%	13.28	%
Average	13.28	%
Stand. Dev.	0.3800	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	10.2	10200
0.6	15.3	7650
1.5	23.5	4700
3	28.8	2880
6	35.7	1785
12	46.3	1157.5
30	65.9	659
60	86.3	431.5
Spindle #	2	

Reference coal grinds from pages 207-10 in notebook 1

TABLE 26 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 160°C 8 Hour Sample

Sample ID:	080604-JH-01	Sample Date:	8/6/2004
Sample Start Time:	0:30	Sample Finish Time:	0:40
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	160ºC	Sample Time:	8 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	147.58	±	0.9257	°C
Reactor Pressure:	118.17	±	15.8347	psig
Flow Rate of Sludge:	532.27	±	2.7977	$gmin^{-1}$
Flow Rate of Caustic:	26.32	±	141.9347	$gmin^{-1}$
Hot Oil Supply Temperature:	199.07	±	2.1694	°C
Hot Oil Return Temperature:	199.59	±	2.0067	°C
Top Flange Temperature:	224.78	±	0.0205	°C
Bottom Flange Temperature:	225.16	±	0.0032	°C
Reactor Level:	5121.24	±	105.4920	mL
Residence Time:	9.58	±	1.6728	min
Caustic Sludge Ratio:	0.0516	±	0.0098	unitless

Percent Solids		
Sample 1 wt%	12.88	%
Sample 1 wt%	12.91	%
Sample 1 wt%	13.18	%
Average	12.99	%
Stand. Dev.	0.1652	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	5	5000
0.6	7.3	3650
1.5	10.3	2060
3	12.5	1250
6	16.6	830
12	22.9	572.5
30	33.5	335
60	43.2	216
Spindle #	2	

Reference coal grinds from pages 211-12 in notebook 1

TABLE 27 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 150°C 2 Hour Sample

Sample ID:	072604-NB-01	Sample Date:	7/26/2004
Sample Start Time:	17:15	Sample Finish Time:	17:25
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	150ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	152.89	±	1.1890	°C
Reactor Pressure:	55.92	±	1.8220	psig
Flow Rate of Sludge:	534.83	±	10.4701	$gmin^{-1}$
Flow Rate of Caustic:	111.40	±	127.9073	$gmin^{-1}$
Hot Oil Supply Temperature:	181.25	±	0.1719	°C
Hot Oil Return Temperature:	181.24	±	0.2371	°C
Top Flange Temperature:	180.04	±	0.0024	°C
Bottom Flange Temperature:	179.83	±	0.0019	°C
Reactor Level:	4845.90	±	282.9894	mL
Residence Time:	7.78	±	1.4437	min
Caustic Sludge Ratio:	0.2189	±	0.0492	unitless

Percent Solids		
Sample 1 wt%	20.82	%
Sample 1 wt%	19.67	%
Sample 1 wt%	20.77	%
Average	20.42	%
Stand. Dev.	0.6500	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	6.4	6400
0.6	8	4000
1.5	10.1	2020
3	12.4	1240
6	15.9	795
12	20.8	520
30	31.1	311
60	43.2	216
Spindle #	2	

Reference coal grinds from pages 237-8 in notebook 1

TABLE 28 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 150°C 4 Hour Sample

Sample ID:	072604-NB-02	Sample Date:	7/26/2004
Sample Start Time:	19:10	Sample Finish Time:	19:20
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	150ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	140.76	±	0.4696	°C
Reactor Pressure:	43.43	±	0.7595	psig
Flow Rate of Sludge:	513.79	±	2.5026	$gmin^{-1}$
Flow Rate of Caustic:	106.24	±	26.6502	$gmin^{-1}$
Hot Oil Supply Temperature:	198.06	±	0.9034	°C
Hot Oil Return Temperature:	198.61	±	1.1902	°C
Top Flange Temperature:	194.23	±	0.3770	°C
Bottom Flange Temperature:	195.21	±	0.4962	°C
Reactor Level:	5255.61	±	84.3742	mL
Residence Time:	8.49	±	0.3767	min
Caustic Sludge Ratio:	0.2072	±	0.0087	unitless

Percent Solids		
Sample 1 wt%	14.61	%
Sample 1 wt%	15.04	%
Sample 1 wt%	14.76	%
Average	14.80	%
Stand. Dev.	0.2183	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	9.3	1860
0.6	12.2	1220
1.5	17.8	712
3	22.8	456
6	29.9	299
12	39.3	196.5
30	56.5	113
60	78.7	78.7
Spindle #	1	

Reference coal grinds from pages 239-40 in notebook 1

TABLE 29 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 150°C 2 Hour Sample

Sample ID:	072704-NB-01	Sample Date:	7/27/2004
Sample Start Time:	15:45	Sample Finish Time:	15:55
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	150ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	148.03	±	0.0000	°C
Reactor Pressure:	93.17	±	0.9935	psig
Flow Rate of Sludge:	500.60	±	2.9221	$gmin^{-1}$
Flow Rate of Caustic:	51.91	±	7.1389	$gmin^{-1}$
Hot Oil Supply Temperature:	180.06	±	0.0178	°C
Hot Oil Return Temperature:	179.89	±	0.0232	°C
Top Flange Temperature:	184.22	±	0.0106	°C
Bottom Flange Temperature:	184.39	±	0.0142	°C
Reactor Level:	5386.61	±	89.3065	mL
Residence Time:	9.75	±	0.2088	min
Caustic Sludge Ratio:	0.1037	±	0.0056	unitless

Percent Solids		
Sample 1 wt%	17.23	%
Sample 1 wt%	17	%
Sample 1 wt%	16.54	%
Average	16.92	%
Stand. Dev.	0.3513	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	7.4	1480
0.6	10.7	1070
1.5	16.5	660
3	21.7	434
6	28.7	287
12	39.1	195.5
30	59	118
60	82	82
Spindle #	1	

Reference coal grinds from pages 241-2 in notebook 1

TABLE 30 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 150°C 4 Hour Sample

Sample ID:	072704-JH-02	Sample Date:	7/27/2004
Sample Start Time:	17:45	Sample Finish Time:	17:55
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	150ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	162.17	±	1.0770	°C
Reactor Pressure:	100.28	±	1.7109	psig
Flow Rate of Sludge:	449.03	±	10.2069	$gmin^{-1}$
Flow Rate of Caustic:	43.67	±	127.6393	$gmin^{-1}$
Hot Oil Supply Temperature:	180.80	±	0.0177	°C
Hot Oil Return Temperature:	180.85	±	0.0231	°C
Top Flange Temperature:	183.78	±	0.0105	°C
Bottom Flange Temperature:	183.80	±	0.0141	°C
Reactor Level:	4106.13	±	580.4286	mL
Residence Time:	9.34	±	4.1629	min
Caustic Sludge Ratio:	0.1025	±	0.0295	unitless

Percent Solids		
Sample 1 wt%	15.99	%
Sample 1 wt%	16.04	%
Sample 1 wt%	15.39	%
Average	15.81	%
Stand. Dev.	0.3617	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	8.9	178
0.6	10.4	104
1.5	14.5	58
3	22.7	45.4
6	36.7	36.7
12	55.9	27.95
30	98.6	19.7
60	---	---
Spindle #	UL	

Reference coal grinds from pages 243-4 in notebook 1

TABLE 31 System Data and Analytical Measurements with 75% Design Basis Sodium Hydroxide Hydrolyzate at 150°C 2 Hour Sample

Sample ID:	072804-NB-01	Sample Date:		7/28/2004
Sample Start Time:	14:26	Sample Finish Time:		14:36
Lysing Agent:	NaOH	Design Basis:		75%
Experiment Temp:	150ºC	Sample Time:		2 hours
System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	151.32	±	0.3744	°C
Reactor Pressure:	62.89	±	0.7151	psig
Flow Rate of Sludge:	526.19	±	1.9928	$gmin^{-1}$
Flow Rate of Caustic:	40.54	±	1.8428	$gmin^{-1}$
Hot Oil Supply Temperature:	200.69	±	0.4357	°C
Hot Oil Return Temperature:	201.50	±	0.4193	°C
Top Flange Temperature:	179.66	±	0.0015	°C
Bottom Flange Temperature:	179.89	±	0.0281	°C
Reactor Level:	4903.38	±	64.3176	mL
Residence Time:	8.65	±	0.1073	min
Caustic Sludge Ratio:	0.0770	±	0.0037	unitless
Percent Solids				
Sample 1 wt%			10.44	%
Sample 1 wt%			10.49	%
Sample 1 wt%			10.2	%
Average			10.38	%
Stand. Dev.			0.1550	%
Viscosity Measurements				
RPM, s^{-1}	Percent Torque, %		Viscosity, cP	
0.3	5.6		1120	
0.6	8.7		870	
1.5	11.4		456	
3	16		320	
6	21.3		213	
12	29.3		146.5	
30	44.3		88.6	
60	61.3		61.3	
Spindle #	1			

Reference coal grinds from pages 245-6 in notebook 1

TABLE 32 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 150°C 2 Hour Sample

Sample ID:	080504-NB-04	Sample Date:	8/5/2004
Sample Start Time:	12:15	Sample Finish Time:	12:25
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	150ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	152.18	±	0.2450	°C
Reactor Pressure:	90.29	±	1.2265	psig
Flow Rate of Sludge:	541.91	±	1.5082	$gmin^{-1}$
Flow Rate of Caustic:	27.78	±	15.5034	$gmin^{-1}$
Hot Oil Supply Temperature:	210.82	±	0.0756	°C
Hot Oil Return Temperature:	211.17	±	0.0366	°C
Top Flange Temperature:	190.14	±	0.0040	°C
Bottom Flange Temperature:	189.96	±	0.0011	°C
Reactor Level:	4790.29	±	66.0546	mL
Residence Time:	8.42	±	0.2550	min
Caustic Sludge Ratio:	0.0513	±	0.0028	unitless

Percent Solids		
Sample 1 wt%	11.02	%
Sample 1 wt%	10.67	%
Sample 1 wt%	11.89	%
Average	11.19	%
Stand. Dev.	0.6282	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	15.2	15200
0.6	21.8	10900
1.5	32.5	6500
3	37.5	3750
6	46.8	2340
12	62.5	1562.5
30	88.1	881
60	---	---
Spindle #	2	

Reference coal grinds from pages 247-8 in notebook 1

TABLE 33 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 150°C 4 Hour Sample

Sample ID:	080504-NB-05	Sample Date:	8/5/2004
Sample Start Time:	14:15	Sample Finish Time:	14:25
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	150ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	152.58	±	0.3958	°C
Reactor Pressure:	91.35	±	2.5225	psig
Flow Rate of Sludge:	537.79	±	2.0534	$gmin^{-1}$
Flow Rate of Caustic:	27.74	±	5.4705	$gmin^{-1}$
Hot Oil Supply Temperature:	212.70	±	0.1435	°C
Hot Oil Return Temperature:	213.45	±	0.0881	°C
Top Flange Temperature:	190.30	±	0.0040	°C
Bottom Flange Temperature:	189.91	±	0.0011	°C
Reactor Level:	4767.35	±	66.3612	mL
Residence Time:	8.43	±	0.1315	min
Caustic Sludge Ratio:	0.0516	±	0.0036	unitless

Percent Solids		
Sample 1 wt%	13.44	%
Sample 1 wt%	13.4	%
Sample 1 wt%	13.43	%
Average	13.42	%
Stand. Dev.	0.0208	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	22.2	22200
0.6	27.8	13900
1.5	39.5	7900
3	48.1	4810
6	62.8	3140
12	75.2	1880
30	84.4	844
60	---	---
Spindle #	2	

Reference coal grinds from pages 249-50 in notebook 1

TABLE 34 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 2 Hour Sample

Sample ID:	080304-JH-01	Sample Date:	8/3/2004
Sample Start Time:	15:05	Sample Finish Time:	15:15
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	140ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	139.31	±	0.0185	°C
Reactor Pressure:	111.43	±	1.8236	psig
Flow Rate of Sludge:	497.15	±	1.2081	$gmin^{-1}$
Flow Rate of Caustic:	101.23	±	33.4243	$gmin^{-1}$
Hot Oil Supply Temperature:	174.96	±	0.0078	°C
Hot Oil Return Temperature:	175.26	±	0.0328	°C
Top Flange Temperature:	140.42	±	0.0056	°C
Bottom Flange Temperature:	140.36	±	0.0057	°C
Reactor Level:	4987.80	±	103.8175	mL
Residence Time:	8.36	±	0.4529	min
Caustic Sludge Ratio:	0.2045	±	0.0132	unitless

Percent Solids		
Sample 1 wt%	13.94	%
Sample 1 wt%	13.7	%
Sample 1 wt%	14.2	%
Average	13.95	%
Stand. Dev.	0.2501	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	20.9	4180
0.6	27.2	2720
1.5	41.5	1660
3	49.1	982
6	57.4	574
12	70.5	352.5
30	95.1	190.2
60	---	---
Spindle #	1	

Reference coal grinds from pages 251-2 in notebook 1

TABLE 35 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 4 Hour Sample

Sample ID:	080304-JH-02	Sample Date:	8/3/2004
Sample Start Time:	17:08	Sample Finish Time:	17:18
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	140ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	141.17	±	0.0801	°C
Reactor Pressure:	124.92	±	0.9793	psig
Flow Rate of Sludge:	464.58	±	1.4217	$gmin^{-1}$
Flow Rate of Caustic:	95.87	±	4.3703	$gmin^{-1}$
Hot Oil Supply Temperature:	175.29	±	0.0078	°C
Hot Oil Return Temperature:	175.82	±	0.0095	°C
Top Flange Temperature:	140.18	±	0.0057	°C
Bottom Flange Temperature:	140.12	±	0.0057	°C
Reactor Level:	5443.43	±	34.5652	mL
Residence Time:	9.71	±	0.0869	min
Caustic Sludge Ratio:	0.2064	±	0.0036	unitless

Percent Solids		
Sample 1 wt%	14.28	%
Sample 1 wt%	15.95	%
Sample 1 wt%	16.31	%
Average	15.51	%
Stand. Dev.	1.0832	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	7.7	7700
0.6	11	5500
1.5	15.8	3160
3	19.6	1960
6	25.5	1275
12	32.6	815
30	44.6	446
60	50.4	252
Spindle #	2	

Reference coal grinds from pages 253-4 in notebook 1

TABLE 36 System Data and Analytical Measurements with 200% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 6 Hour Sample

Sample ID:	080304-JH-03	Sample Date:	8/3/2004
Sample Start Time:	19:03	Sample Finish Time:	19:13
Lysing Agent:	NaOH	Design Basis:	200%
Experiment Temp:	140ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	141.67	±	0.0376	°C
Reactor Pressure:	123.46	±	1.2827	psig
Flow Rate of Sludge:	465.41	±	1.2951	$gmin^{-1}$
Flow Rate of Caustic:	95.67	±	4.4404	$gmin^{-1}$
Hot Oil Supply Temperature:	175.60	±	0.0078	°C
Hot Oil Return Temperature:	176.20	±	0.0095	°C
Top Flange Temperature:	140.06	±	0.0005	°C
Bottom Flange Temperature:	139.55	±	0.0311	°C
Reactor Level:	5429.46	±	47.2410	mL
Residence Time:	9.68	±	0.0948	min
Caustic Sludge Ratio:	0.2056	±	0.0035	unitless
Percent Solids				
Sample 1 wt%			16.78	%
Sample 1 wt%			16.46	%
Sample 1 wt%			15.48	%
Average			16.24	%
Stand. Dev.			0.6773	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	18.1	3620
0.6	23	2300
1.5	21.4	856
3	40.9	818
6	53.3	533
12	69.5	347.5
30	99.2	198.4
60	---	---
Spindle #	1	

Reference coal grinds from pages 255-6 in notebook 1

TABLE 37 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 2 Hour Sample

Sample ID:	080404-NB-01	Sample Date:	8/4/2004
Sample Start Time:	12:15	Sample Finish Time:	12:25
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	140ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	143.04	±	0.0398	°C
Reactor Pressure:	48.84	±	0.3041	psig
Flow Rate of Sludge:	516.09	±	1.5902	$gmin^{-1}$
Flow Rate of Caustic:	52.92	±	2.7544	$gmin^{-1}$
Hot Oil Supply Temperature:	184.49	±	0.0452	°C
Hot Oil Return Temperature:	183.93	±	0.0259	°C
Top Flange Temperature:	181.22	±	0.0129	°C
Bottom Flange Temperature:	180.46	±	0.0092	°C
Reactor Level:	4317.58	±	57.5266	mL
Residence Time:	7.59	±	0.1128	min
Caustic Sludge Ratio:	0.1025	±	0.0030	unitless

Percent Solids		
Sample 1 wt%	13.63	%
Sample 1 wt%	12.5	%
Sample 1 wt%	13.29	%
Average	13.14	%
Stand. Dev.	0.5797	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	5.3	5300
0.6	7.2	3600
1.5	9.7	1940
3	12.8	1280
6	16.6	830
12	23	575
30	34.5	345
60	46.5	232.5
Spindle #	2	

Reference coal grinds from pages 257-8 in notebook 1

TABLE 38 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 4 Hour Sample

Sample ID:	080404-NB-02	Sample Date:	8/4/2004
Sample Start Time:	14:30	Sample Finish Time:	14:40
Lysing Agent:	NaOH	Design Basis:	100%
Experiment Temp:	140ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	137.77	±	0.1047	°C
Reactor Pressure:	49.96	±	0.5424	psig
Flow Rate of Sludge:	514.72	±	2.0230	$gmin^{-1}$
Flow Rate of Caustic:	53.37	±	6.6908	$gmin^{-1}$
Hot Oil Supply Temperature:	183.72	±	0.0236	°C
Hot Oil Return Temperature:	183.91	±	0.0458	°C
Top Flange Temperature:	180.62	±	0.0129	°C
Bottom Flange Temperature:	180.89	±	0.0092	°C
Reactor Level:	4535.35	±	62.7395	mL
Residence Time:	7.99	±	0.1348	min
Caustic Sludge Ratio:	0.1037	±	0.0033	unitless

Percent Solids		
Sample 1 wt%	14.3	%
Sample 1 wt%	13.75	%
Sample 1 wt%	14.61	%
Average	14.22	%
Stand. Dev.	0.4355	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	8	8000
0.6	11.2	5600
1.5	15.3	3060
3	18.1	1810
6	24.5	1225
12	32.8	820
30	47.2	472
60	63.1	315.5
Spindle #	2	

Reference coal grinds from pages 259-60 in notebook 1

TABLE 39 System Data and Analytical Measurements with 100% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 6 Hour Sample

Sample ID:	080404-NB-03	Sample Date:	8/4/2004
Sample Start Time:	15:03	Sample Finish Time:	15:13
Lysing Agent:	NaOH	Design Basis:	1 X
Experiment Temp:	140	Sample Time:	6

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	139.59	±	0.0560	°C
Reactor Pressure:	52.53	±	0.7086	psig
Flow Rate of Sludge:	515.05	±	2.3815	$gmin^{-1}$
Flow Rate of Caustic:	52.66	±	3.7760	$gmin^{-1}$
Hot Oil Supply Temperature:	188.72	±	0.0285	°C
Hot Oil Return Temperature:	188.98	±	0.0527	°C
Top Flange Temperature:	190.87	±	0.0138	°C
Bottom Flange Temperature:	189.30	±	0.0102	°C
Reactor Level:	4279.79	±	81.7572	mL
Residence Time:	7.54	±	0.1325	min
Caustic Sludge Ratio:	0.1022	±	0.0042	unitless

Percent Solids		
Sample 1 wt%	14.55	%
Sample 1 wt%	14.77	%
Sample 1 wt%	14.71	%
Average	14.68	%
Stand. Dev.	0.1137	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	6.2	6200
0.6	8.5	4250
1.5	12.7	2540
3	16.3	1630
6	21.6	1080
12	29.1	727.5
30	42.4	424
60	63.1	315.5
Spindle #	2	

Reference coal grinds from pages 261-62 in notebook 1

TABLE 40 System Data and Analytical Measurements with 75% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 2 Hour Sample

Sample ID:	080404-JH-04	Sample Date:	8/4/2004
Sample Start Time:	20:20	Sample Finish Time:	20:30
Lysing Agent:	NaOH	Design Basis:	75%
Experiment Temp:	140ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	146.74	±	2.1178	°C
Reactor Pressure:	127.47	±	3.1802	psig
Flow Rate of Sludge:	525.63	±	2.8783	$gmin^{-1}$
Flow Rate of Caustic:	41.05	±	20.8729	$gmin^{-1}$
Hot Oil Supply Temperature:	189.30	±	0.0046	°C
Hot Oil Return Temperature:	189.78	±	0.0021	°C
Top Flange Temperature:	190.05	±	0.0005	°C
Bottom Flange Temperature:	189.90	±	0.0000	°C
Reactor Level:	4455.79	±	177.3643	mL
Residence Time:	7.87	±	0.4028	min
Caustic Sludge Ratio:	0.0781	±	0.0040	unitless
Percent Solids				
Sample 1 wt%			13.86	%
Sample 1 wt%			13.92	%
Sample 1 wt%			14.04	%
Average			13.94	%
Stand. Dev.			0.0917	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	16	3200
0.6	21	2100
1.5	27.1	1084
3	34.9	698
6	47.4	474
12	62.8	314
30	92.2	184.4
60	---	---
Spindle #	1	

Reference coal grinds from pages 263-4 in notebook 1

TABLE 41 System Data and Analytical Measurements with 75% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 4 Hour Sample

Sample ID:	080404-JH-05	Sample Date:	8/4/2004
Sample Start Time:	22:30	Sample Finish Time:	22:40
Lysing Agent:	NaOH	Design Basis:	75%
Experiment Temp:	140ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	149.48	±	2.9356	°C
Reactor Pressure:	120.71	±	4.5967	psig
Flow Rate of Sludge:	351.00	±	6.9983	$gmin^{-1}$
Flow Rate of Caustic:	29.47	±	163.9266	$gmin^{-1}$
Hot Oil Supply Temperature:	189.51	±	0.0046	°C
Hot Oil Return Temperature:	189.87	±	0.0021	°C
Top Flange Temperature:	190.03	±	0.0005	°C
Bottom Flange Temperature:	189.90	±	0.0000	°C
Reactor Level:	5044.40	±	364.5895	mL
Residence Time:	16.02	±	7.0126	min
Caustic Sludge Ratio:	0.0982	±	0.0420	unitless
Percent Solids				
Sample 1 wt%			13.63	%
Sample 1 wt%			13.92	%
Sample 1 wt%			13.37	%
Average			13.64	%
Stand. Dev.			0.2751	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	5.8	5800
0.6	9	4500
1.5	12.5	2500
3	15.2	1520
6	19.9	995
12	26.6	665
30	39	390
60	51.8	259
Spindle #	2	

Reference coal grinds from pages 264-5 in notebook 1

TABLE 42 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 2 Hour Sample

Sample ID:	080504-JH-01	Sample Date:	8/5/2004
Sample Start Time:	4:00	Sample Finish Time:	4:10
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	140ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	151.84	±	0.2346	°C
Reactor Pressure:	96.49	±	3.5439	psig
Flow Rate of Sludge:	516.06	±	2.6509	$gmin^{-1}$
Flow Rate of Caustic:	26.96	±	11.3213	$gmin^{-1}$
Hot Oil Supply Temperature:	181.03	±	0.0353	°C
Hot Oil Return Temperature:	181.64	±	0.0345	°C
Top Flange Temperature:	189.99	±	0.0005	°C
Bottom Flange Temperature:	189.91	±	0.0005	°C
Reactor Level:	5472.98	±	231.9710	mL
Residence Time:	10.08	±	0.4443	min
Caustic Sludge Ratio:	0.0522	±	0.0050	unitless
Percent Solids				
Sample 1 wt%			14.21	%
Sample 1 wt%			14.42	%
Sample 1 wt%			14.42	%
Average			14.35	%
Stand. Dev.			0.1212	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	11	11000
0.6	15.1	7550
1.5	22.7	4540
3	28.9	2890
6	37.6	1880
12	49.3	1232.5
30	71.9	719
60	94.4	472
Spindle #	2	

Reference coal grinds from pages 267-8 in notebook 1

TABLE 43 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 4 Hour Sample

Sample ID:	080504-JH-02	Sample Date:	8/5/2004
Sample Start Time:	6:00	Sample Finish Time:	6:10
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	140ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	143.48	±	0.1317	°C
Reactor Pressure:	88.77	±	3.7157	psig
Flow Rate of Sludge:	491.15	±	7.2969	$gmin^{-1}$
Flow Rate of Caustic:	23.61	±	62.1823	$gmin^{-1}$
Hot Oil Supply Temperature:	184.06	±	0.0079	°C
Hot Oil Return Temperature:	184.58	±	0.0085	°C
Top Flange Temperature:	190.01	±	0.0005	°C
Bottom Flange Temperature:	189.93	±	0.0005	°C
Reactor Level:	5907.46	±	151.0911	mL
Residence Time:	11.81	±	2.4747	min
Caustic Sludge Ratio:	0.0465	±	0.0132	unitless
Percent Solids				
Sample 1 wt%			12.98	%
Sample 1 wt%			12.69	%
Sample 1 wt%			12.46	%
Average			12.71	%
Stand. Dev.			0.2606	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	20.8	20800
0.6	26.5	13250
1.5	37.7	7540
3	43	4300
6	59	2950
12	74.6	1865
30	---	---
60	---	---
Spindle #	2	

Reference coal grinds from pages 269-70 in notebook 1

TABLE 44 System Data and Analytical Measurements with 50% Design Basis Sodium Hydroxide Hydrolyzate at 140°C 6 Hour Sample

Sample ID:	080504-JH-03	Sample Date:	8/5/2004
Sample Start Time:	8:00	Sample Finish Time:	8:10
Lysing Agent:	NaOH	Design Basis:	50%
Experiment Temp:	140ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	159.64	±	1.9166	°C
Reactor Pressure:	95.05	±	7.0915	psig
Flow Rate of Sludge:	172.66	±	13.3697	$gmin^{-1}$
Flow Rate of Caustic:	12.15	±	296.7273	$gmin^{-1}$
Hot Oil Supply Temperature:	183.73	±	0.0079	°C
Hot Oil Return Temperature:	184.22	±	0.0084	°C
Top Flange Temperature:	190.03	±	0.0005	°C
Bottom Flange Temperature:	189.95	±	0.0005	°C
Reactor Level:	4149.86	±	549.5543	mL
Residence Time:	-106.89	±	1284.5764	min
Caustic Sludge Ratio:	-0.0154	±	1.9239	unitless
Percent Solids				
Sample 1 wt%				%
Sample 1 wt%				%
Sample 1 wt%				%
Average				%
Stand. Dev.				%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
0.3	5.5	5500
0.6	7.3	3650
1.5	10.1	2020
3	13	1300
6	16.9	845
12	22.4	560
30	32.1	321
60	42.6	213
Spindle #	2	

Reference coal grinds from pages in notebook 1

TABLE 45 System Data and Analytical Measurements with 100% Design Basis Sulfuric Acid Hydrolyzate at 160°C 2 Hour Sample

Sample ID:	081104-JH-01-H2SO4	Sample Date:	8/11/2004
Sample Start Time:	16:20	Sample Finish Time:	16:30
Lysing Agent:	H_2SO_4	Design Basis:	100%
Experiment Temp:	160ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	160.91	±	0.3574	°C
Reactor Pressure:	158.37	±	26.8773	psig
Flow Rate of Sludge:	451.65	±	3.0992	$gmin^{-1}$
Flow Rate of Caustic:	25.44	±	5.3041	$gmin^{-1}$
Hot Oil Supply Temperature:	211.49	±	0.0148	°C
Hot Oil Return Temperature:	212.37	±	0.0076	°C
Top Flange Temperature:	179.77	±	0.0017	°C
Bottom Flange Temperature:	179.44	±	0.0060	°C
Reactor Level:	4691.06	±	176.7279	mL
Residence Time:	9.83	±	0.3625	min
Caustic Sludge Ratio:	0.0563	±	0.0063	unitless

Percent Solids		
Sample 1 wt%	11.4	%
Sample 1 wt%	12.13	%
Sample 1 wt%	11.81	%
Average	11.78	%
Stand. Dev.	0.3659	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
Spindle #	N/A	

Reference coal grinds from pages 271-2 in notebook 1

TABLE 46 System Data and Analytical Measurements with 100% Design Basis Sulfuric Acid Hydrolyzate at 160°C 4 Hour Sample

Sample ID:	081104-JH-02-H2SO4	Sample Date:	8/11/2004
Sample Start Time:	18:25	Sample Finish Time:	18:35
Lysing Agent:	H_2SO_4	Design Basis:	100%
Experiment Temp:	160ºC	Sample Time:	4 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	154.89	±	0.4810	°C
Reactor Pressure:	141.00	±	29.1845	psig
Flow Rate of Sludge:	439.71	±	2.4947	$gmin^{-1}$
Flow Rate of Caustic:	23.31	±	23.2720	$gmin^{-1}$
Hot Oil Supply Temperature:	212.12	±	0.0148	°C
Hot Oil Return Temperature:	212.69	±	0.0076	°C
Top Flange Temperature:	179.84	±	0.0017	°C
Bottom Flange Temperature:	179.19	±	0.0060	°C
Reactor Level:	5006.34	±	193.0660	mL
Residence Time:	10.84	±	0.6716	min
Caustic Sludge Ratio:	0.0532	±	0.0066	unitless

Percent Solids		
Sample 1 wt%	10.7	%
Sample 1 wt%	11.01	%
Sample 1 wt%	11.41	%
Average	11.04	%
Stand. Dev.	0.3559	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
Spindle #	N/A	

Reference coal grinds from pages 273-4 in notebook 1

TABLE 47 System Data and Analytical Measurements with 100% Design Basis Sulfuric Acid Hydrolyzate at 160°C 6 Hour Sample

Sample ID:	081104-JH-03-H2SO4	Sample Date:	8/11/2004
Sample Start Time:	20:45	Sample Finish Time:	20:55
Lysing Agent:	H_2SO_4	Design Basis:	100%
Experiment Temp:	160ºC	Sample Time:	6 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	153.79	±	1.6674	°C
Reactor Pressure:	96.28	±	2.8544	psig
Flow Rate of Sludge:	385.29	±	4.4212	$gmin^{-1}$
Flow Rate of Caustic:	18.96	±	44.7613	$gmin^{-1}$
Hot Oil Supply Temperature:	220.77	±	0.0183	°C
Hot Oil Return Temperature:	221.40	±	0.0214	°C
Top Flange Temperature:	179.93	±	0.0017	°C
Bottom Flange Temperature:	179.82	±	0.0135	°C
Reactor Level:	5493.53	±	151.5063	mL
Residence Time:	13.95	±	3.0719	min
Caustic Sludge Ratio:	0.0489	±	0.0086	unitless

Percent Solids		
Sample 1 wt%	9.92	%
Sample 1 wt%	9.68	%
Sample 1 wt%	10.45	%
Average	10.02	%
Stand. Dev.	0.3940	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
Spindle #	N/A	

Reference coal grinds from pages 275-6 in notebook 1

TABLE 48 System Data and Analytical Measurements with 50% Design Basis Sulfuric Acid Hydrolyzate at 160°C 2 Hour Sample

Sample ID:	081204-NB-01-H2SO4	Sample Date:	8/12/2004
Sample Start Time:	0:00	Sample Finish Time:	0:10
Lysing Agent:	H_2SO_4	Design Basis:	50%
Experiment Temp:	160ºC	Sample Time:	2 hours

System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	160.88	±	0.1248	^oC
Reactor Pressure:	163.85	±	3.0019	psig
Flow Rate of Sludge:	273.65	±	1.1929	$gmin^{-1}$
Flow Rate of Caustic:	7.10	±	5.5470	$gmin^{-1}$
Hot Oil Supply Temperature:	222.01	±	0.0184	^oC
Hot Oil Return Temperature:	222.85	±	0.0215	^oC
Top Flange Temperature:	219.59	±	0.0012	^oC
Bottom Flange Temperature:	219.20	±	0.0073	^oC
Reactor Level:	5567.93	±	70.4130	mL
Residence Time:	19.84	±	0.4006	min
Caustic Sludge Ratio:	0.0259	±	0.0042	unitless

Percent Solids		
Sample 1 wt%	10.86	%
Sample 1 wt%	10.54	%
Sample 1 wt%	10.75	%
Average	10.72	%
Stand. Dev.	0.1626	%

Viscosity Measurements		
RPM, s^{-1}	Percent Torque, %	Viscosity, cP
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
N/A	N/A	N/A
Spindle #	N/A	

Reference coal grinds from pages 277-8 in notebook 1

TABLE 49 System Data and Analytical Measurements with 50% Design Basis Sulfuric Acid Hydrolyzate at 160°C 4 Hour Sample

Sample ID:	081204-NB-02-H2SO4	Sample Date:		8/12/2004
Sample Start Time:	2:00	Sample Finish Time:		2:10
Lysing Agent:	H_2SO_4	Design Basis:		50%
Experiment Temp:	160ºC	Sample Time:		4 hours
System Conditions at Time of Sampling (Averaging over 10 minutes)				
	Average	±	Std. Dev.	Units
Reactor Temperature:	161.24	±	0.0943	°C
Reactor Pressure:	147.17	±	3.6378	psig
Flow Rate of Sludge:	284.77	±	1.2218	$gmin^{-1}$
Flow Rate of Caustic:	7.56	±	2.2416	$gmin^{-1}$
Hot Oil Supply Temperature:	225.30	±	0.1014	°C
Hot Oil Return Temperature:	225.71	±	0.0881	°C
Top Flange Temperature:	219.64	±	0.0012	°C
Bottom Flange Temperature:	219.22	±	0.3828	°C
Reactor Level:	5511.40	±	48.9790	mL
Residence Time:	18.85	±	0.1964	min
Caustic Sludge Ratio:	0.0265	±	0.0042	unitless
Percent Solids				
Sample 1 wt%	10.28			%
Sample 1 wt%	10.03			%
Sample 1 wt%	11.07			%
Average	10.46			%
Stand. Dev.	0.5429			%
Viscosity Measurements				
RPM, s^{-1}	Percent Torque, %		Viscosity, cP	
N/A	N/A		N/A	
N/A	N/A		N/A	
N/A	N/A		N/A	
N/A	N/A		N/A	
N/A	N/A		N/A	
N/A	N/A		N/A	
N/A	N/A		N/A	
N/A	N/A		N/A	
Spindle #	N/A			

Reference coal grinds from pages 279-80 in notebook 1

APPENDIX B VALVE CONFIGURATIONS USED FOR OPERATION OF UNIT BX ILLUSTRATIONS

APPENDIX B LIST OF SUPPLEMENTRY FIGURES

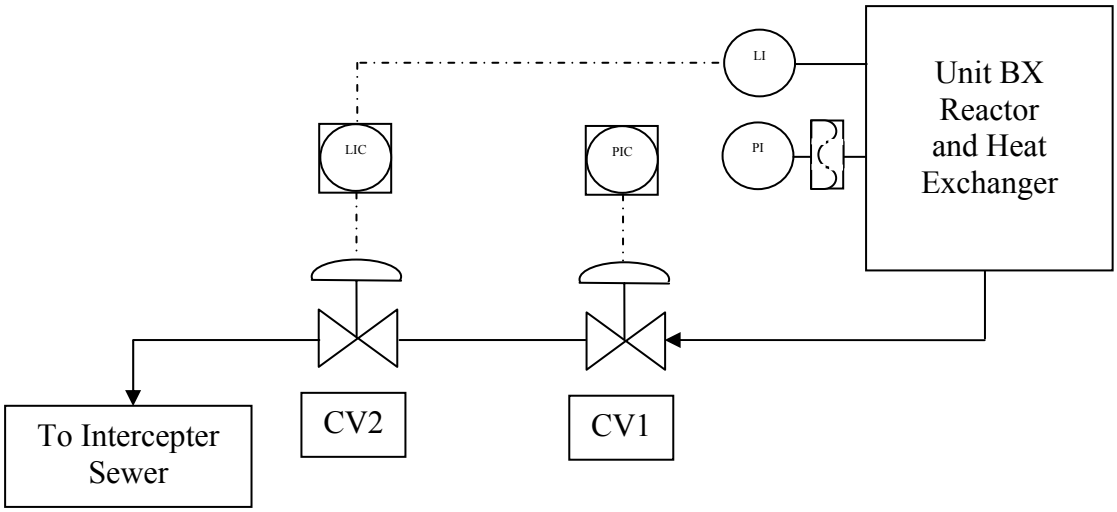

FIGURE 1 Level Control Using CV2 Only in the Horizontal Orientation

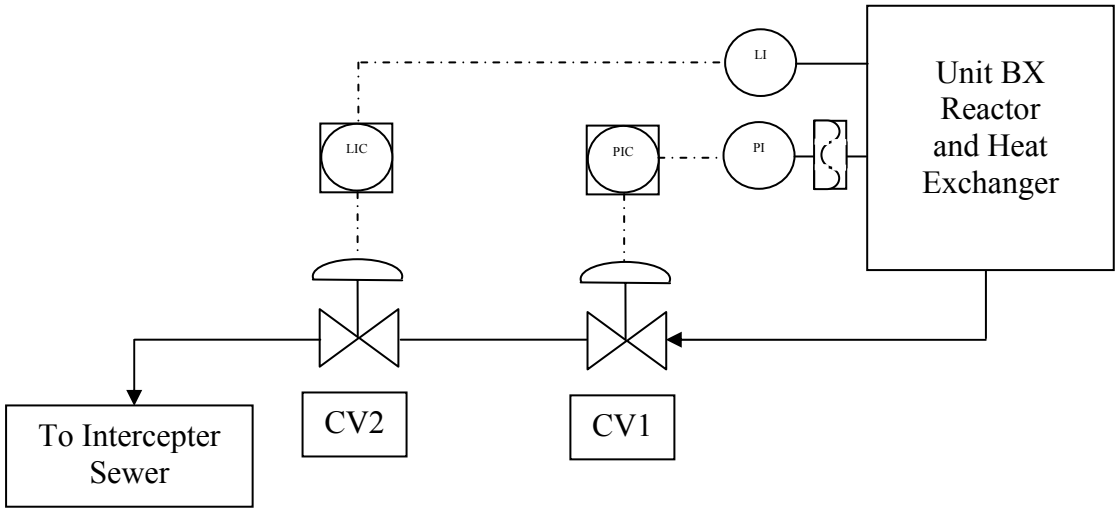

FIGURE 2 Level Control Using CV1 to control pressure and CV2 for Controlling Level

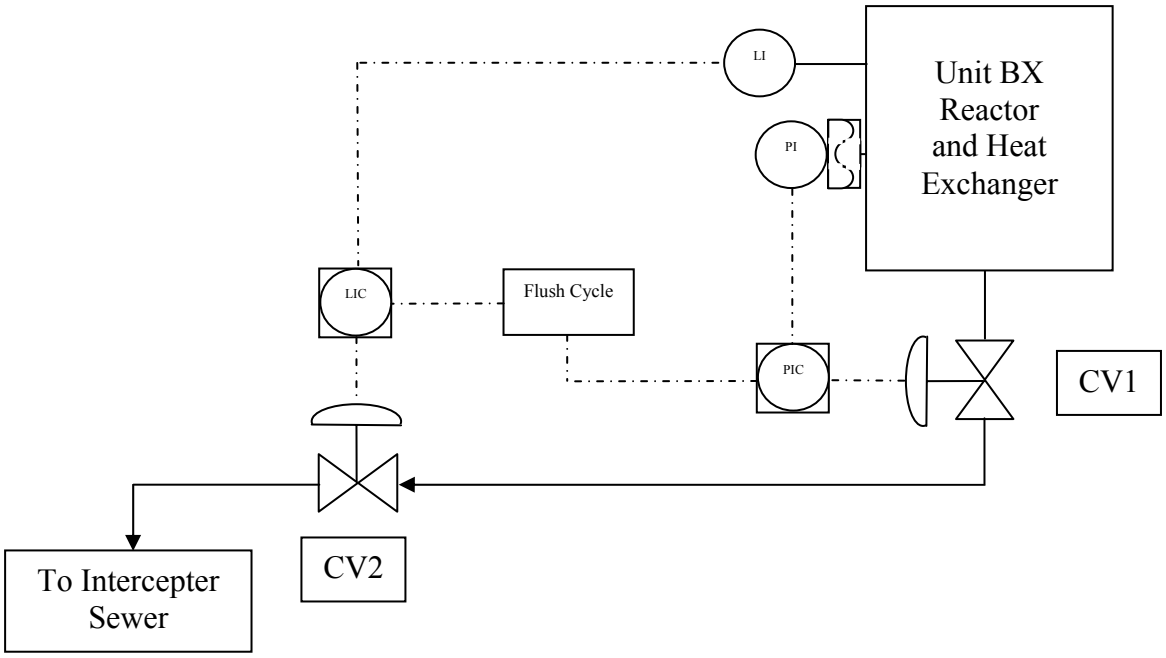

FIGURE 3 ***Level Control with CV1 in the Vertical Orientation After the Heat Exchanger and CV2 in the Horizontal Orientation Pressure with a Flush Cycle***

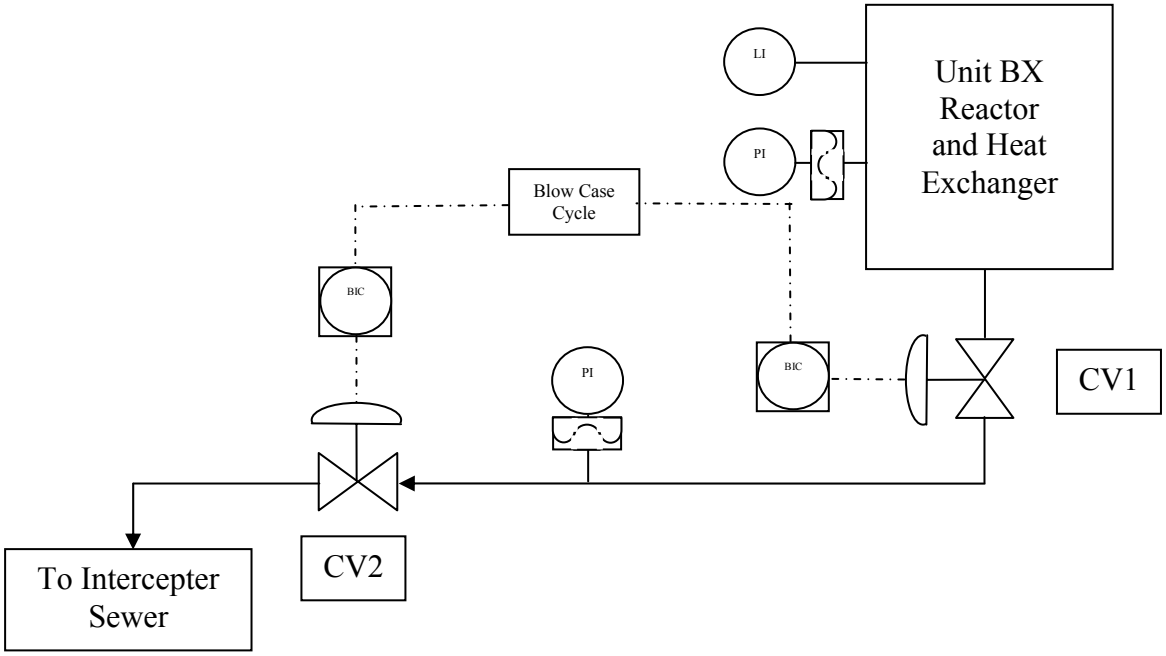

FIGURE 4 ***Blow Case with CV1 in the Vertical Orientation and CV2 in the Horizontal Orientation***

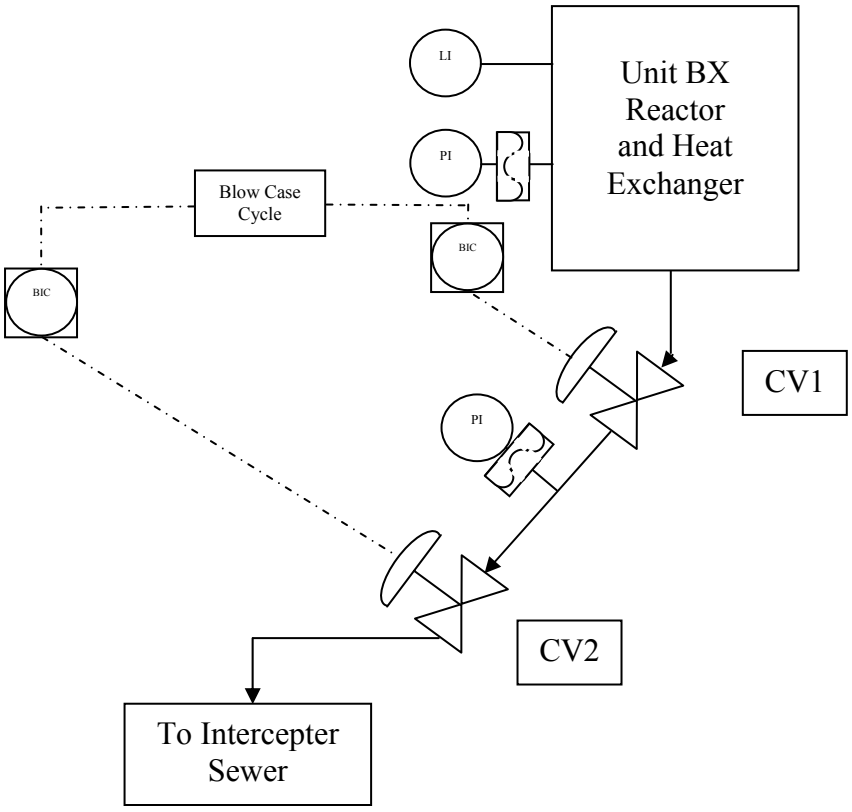

FIGURE 5 Blow Case with CV1 and CV2 at a 45º incline

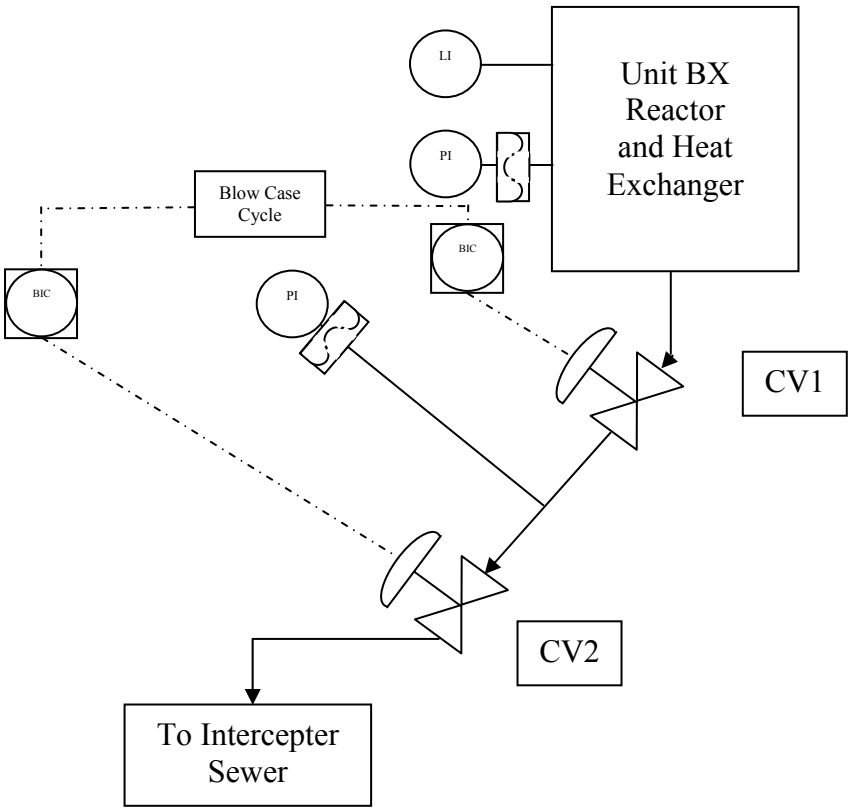

FIGURE 6 Blow Case with CV1 and CV2 at a 45º Incline with the Addition of a Large Stand Pipe

APPENDIX C COAL SLURRY TEST DATA

APPENDIX C LIST OF SUPPLIMENTRY TABLES

TABLE 1 Calculated Coal Slurry Make-Up for Tests at 170 °C

Coal Sludge Recipe										
		Sample ID	Hour Sample	%water	%Coal	%Sludge Water	%Sludge Solids	%Defoamer	%ALS	Calculated Solids
170 °C	200%	72104-JH-01	2	16.00%	58.88%	16.00%	9.12%	0.00%	0.00%	68.00%
		72104-JH-01-redo	2	16.00%	61.58%	16.00%	6.42%	0.00%	0.00%	68.00%
		72104-JH-02	4	16.00%	63.32%	16.00%	4.68%	0.00%	0.00%	68.00%
		72104-JH-03	6	16.00%	63.01%	16.00%	4.99%	0.00%	0.00%	68.00%
		72104-JH-04	8	16.00%	63.47%	16.00%	4.53%	0.00%	0.00%	68.00%
	50%	72204-NB-01	2	16.00%	62.86%	16.00%	5.14%	0.00%	0.00%	68.00%
		72204-NB-02	4	16.00%	61.08%	16.00%	6.92%	0.00%	0.00%	68.00%
		72204-NB-03	6	16.00%	65.43%	16.00%	2.57%	0.00%	0.00%	68.00%
		72204-JH-04	8	16.00%	65.32%	16.00%	2.68%	0.00%	0.00%	68.00%
	75%	072304-JH-01	2	16.00%	67.15%	16.00%	0.85%	0.00%	0.00%	68.00%
		072304-JH-02	4	16.00%	66.40%	16.00%	1.60%	0.00%	0.00%	68.00%
	50%	070804-NB-01	2	16.00%	65.73%	16.00%	2.27%	0.00%	0.00%	68.00%
		070804-NB-02	4	16.00%	65.71%	16.00%	2.29%	0.00%	0.00%	68.00%

TABLE 2 Percent Solids in Coal Slurry Samples for Tests at 170 °C

Weight Percent of Slurry Total Solids								
		Sample ID	Hour Sample	wt%1	wt%2	wt%3	slurry wt%avg	slurry wt%avg Stand. Dev
170 °C	200%	72104-JH-01	2	66.29	66.27	66.32	66.29	0.0252
		72104-JH-01-redo	2	66.58	66.58	66.21	66.46	0.2136
		72104-JH-02	4	68.01	67.99	67.84	67.95	0.0929
		72104-JH-03	6	68.00	68.00	68.00	68.00	0.0000
		72104-JH-04	8	68.00	68.00	68.00	68.00	0.0000
	100%	72204-NB-01	2	65.90	65.94	66.10	65.98	0.1058
		72204-NB-02	4	64.60	64.52	64.49	64.54	0.0569
		72204-NB-03	6	67.23	67.36	67.43	67.34	0.1015
		72204-JH-04	8	65.83	65.86	65.82	65.84	0.0208
	75%	072304-JH-01	2	69.53	69.21	69.28	69.34	0.1682
		072304-JH-02	4	68.50	68.85	69.00	68.78	0.2566
	50%	070804-NB-01	2	68.52		68.67	68.60	0.1061
		070804-NB-02	4	67.48	67.19	67.23	67.30	0.1572

TABLE 3 Results of Coal Slurry Test, pH for Samples at 170 ^{o}C

pH							
		Sample ID	Hour Sample	pH	pOH	Hydrogen Ion Concentration, molL^{-1}	Hydroxyl Ion Concentration, molL^{-1}
170 ^{o}C	200%	72104-JH-01	2	13.72	0.28	1.905E-14	0.5248075
		72104-JH-01-redo	2	13.51	0.49	3.09E-14	0.3235937
		72104-JH-02	4	13.63	0.37	2.344E-14	0.4265795
		72104-JH-03	6	13.3	0.7	5.012E-14	0.1995262
		72104-JH-04	8	13.33	0.67	4.677E-14	0.2137962
	100%	72204-NB-01	2	12.87	1.13	1.349E-13	0.074131
		72204-NB-02	4	12.96	1.04	1.096E-13	0.0912011
		72204-NB-03	6	12.88	1.12	1.318E-13	0.0758578
		72204-JH-04	8	12.7	1.3	1.995E-13	0.0501187
	75%	072304-JH-01	2	12.49	1.51	3.236E-13	0.030903
		072304-JH-02	4	12.7	1.3	1.995E-13	0.0501187
	50%	070804-NB-01	2	13.26	0.74	5.495E-14	0.1819701
		070804-NB-02	4	13.25	0.75	5.623E-14	0.1778279

TABLE 4 Results of Coal Slurry Test, Viscosities of Samples from Tests at 170 °C

Viscosity											
		Sample ID	Hour Sample	Initial Viscosity	Initial Torque	Initial RPM	Final Viscosity	Final torque	Final RPM	Spindle	Comment
170 °C	200%	72104-JH-01	2	11250	85.2	3	7227	54.2	3	2	
		72104-JH-01-redo	2	20320	76.2	1.5	13330	50	1.5	2	
		72104-JH-02	4	13307	99.8	3	8960	67.2	3	2	
		72104-JH-03	6	64000	96	0.6	59600	89.3	0.6	2	
		72104-JH-04	8	49800	74.7	0.6	45333	68.3	0.6	2	
	100%	72204-NB-01	2	57067	84.1	0.6	35133	52.8	0.6	2	foamy
		72204-NB-02	4	54000	81	0.6	36533	54.8	0.6	2	
		72204-NB-03	6	1067	75.5	3	8107	60.8	3	2	foamy
		72204-JH-04	8	19600	73.5	1.5	11213	84.1	3	2	foamy
	75%	072304-JH-01	2	11000	81.5	3	9600	72	3	2	
		072304-JH-02	4	10400	78	3	7613	57.2	3	2	
	50%	070804-NB-01	2	11920	89.4	3	8853	66.4	3	2	
		070804-NB-02	4	12147	91.1	3	11253	84.4	3	2	

TABLE 5 Coal Slurry Results, Raw Particle Size Distribution for Tests at 170 ^{o}C

Particle Size Distribution								
		Sample ID	Hour Sample	20	40	100	200	325
170 °C	200%	72104-JH-01	2	457.03	409.03	409.34	351.21	336.39
		72104-JH-01-redo	2	456.99	408.98	398.87	356.67	339.76
		72104-JH-02	4	456.98	409.05	395.40	374.86	335.48
		72104-JH-03	6	456.48		399.99	352.08	335.69
		72104-JH-04	8	390.99	361.90	363.91	334.54	315.50
	100%	72204-NB-01	2	457.68	409.75	426.57	341.87	335.22
		72204-NB-02	4	391.49	361.67	351.45	332.66	319.00
		72204-NB-03	6	456.76	409.65	402.39	347.46	339.42
		72204-JH-04	8	392.01	362.61	361.67	337.87	317.81
	75%	072304-JH-01	2	456.70	410.26	401.89	345.72	337.03
		072304-JH-02	4	390.42	363.11	354.02	337.34	320.55
	50%	070804-NB-01	2	456.51	410.35	396.31	349.55	336.60
		070804-NB-02	4	390.93	361.82	356.25	344.48	317.20

TABLE 6 Sieves Used for Specific Coal Grind Tests at 170 °C

Particle Size Distribution									
				Standard Weight					
		Sample ID	Hour Sample	Set	20	40	100	200	325
170 °C	200%	72104-JH-01	2	Sieve 1	456.39	408.51	375.29	335.49	331.27
		72104-JH-01-redo	2	Sieve 1	456.39	408.51	375.29	335.49	331.27
		72104-JH-02	4	Sieve 1	456.39	408.51	375.29	335.49	331.27
		72104-JH-03	6	Sieve 1	456.39	408.51	375.29	335.49	331.27
		72104-JH-04	8	Sieve 2	390.22	361.28	331.32	325.24	312.32
	100%	72204-NB-01	2	Sieve 1	456.39	408.51	375.29	335.49	331.27
		72204-NB-02	4	Sieve 2	390.22	361.28	331.32	325.24	312.32
		72204-NB-03	6	Sieve1	456.39	408.51	375.29	335.49	331.27
		72204-JH-04	8	Sieve 2	390.22	361.28	331.32	325.24	312.32
	75%	072304-JH-01	2	Sieve 1	456.39	408.51	375.29	335.49	331.27
		072304-JH-02	4	Sieve 2	390.22	361.28	331.32	325.24	312.32
	50%	070804-NB-01	2	Sieve 1	456.39	408.51	375.29	335.49	331.27
		070804-NB-02	4	Sieve 2	390.22	361.28	331.32	325.24	312.32

TABLE 7 Coal Slurry Results, Difference Particle Size Distribution for Tests at 170 °C

Particle Size Distribution											
				Difference							
		Sample ID	Hour Sample	20	40	100	200	325	Total Solids in Sieves	Total Solids of Slurry	>325 Mesh
170 °C	200%	72104-JH-01	2	0.64	0.52	34.05	15.72	5.12	56.05	66.29	10.24
		72104-JH-01-redo	2	0.60	0.47	23.58	21.18	8.49	54.32	66.46	12.14
		72104-JH-02	4	0.59	0.54	20.11	39.37	4.21	64.82	67.95	3.13
		72104-JH-03	6	0.09	0.00	24.70	16.59	4.42	45.80	68.00	22.20
		72104-JH-04	8	0.77	0.62	32.59	9.30	3.18	46.46	68.00	21.54
	100%	72204-NB-01	2	1.29	1.24	51.28	6.38	3.95	64.14	65.98	1.84
		72204-NB-02	4	1.27	0.39	20.13	7.42	6.68	35.89	64.54	28.65
		72204-NB-03	6	0.37	1.14	27.10	11.97	8.15	48.73	67.34	18.61
		72204-JH-04	8	1.79	1.33	30.35	12.63	5.49	51.59	65.84	14.25
	75%	072304-JH-01	2	0.31	1.75	26.60	10.23	5.76	44.65	69.34	24.69
		072304-JH-02	4	0.20	1.83	22.70	12.10	8.23	45.06	68.78	23.72
	50%	070804-NB-01	2	0.12	1.84	21.02	14.06	5.33	42.37	68.60	26.22
		070804-NB-02	4	0.71	0.54	24.93	19.24	4.88	50.30	67.30	17.00

TABLE 8 Coal Slurry Results, Normalized Particle Size Distribution for Tests at 170 °C

Particle Size Distribution									
				Normalized					
		Sample ID	Hour Sample	20	40	100	200	325	>325 mesh size
170 °C	200%	72104-JH-01	2	0.009654	0.007844	0.513626	0.237128	0.077233	0.154515
		72104-JH-01-redo	2	0.009028	0.007072	0.354818	0.318704	0.127752	0.182625
		72104-JH-02	4	0.008683	0.007947	0.295967	0.579425	0.061960	0.046016
		72104-JH-03	6	0.001324	0.000000	0.363235	0.243971	0.065000	0.326471
		72104-JH-04	8	0.011324	0.009118	0.479265	0.136765	0.046765	0.316765
	100%	72204-NB-01	2	0.019551	0.018794	0.777205	0.096696	0.059867	0.027887
		72204-NB-02	4	0.019679	0.006043	0.311916	0.114973	0.103507	0.443882
		72204-NB-03	6	0.005495	0.016929	0.402435	0.177755	0.121028	0.276359
		72204-JH-04	8	0.027188	0.020202	0.460989	0.191838	0.083388	0.216394
	75%	072304-JH-01	2	0.004471	0.025238	0.383617	0.147534	0.083069	0.356072
		072304-JH-02	4	0.002908	0.026605	0.330022	0.175915	0.119651	0.344899
	50%	070804-NB-01	2	0.001749	0.026824	0.306436	0.204971	0.077702	0.382316
		070804-NB-02	4	0.010550	0.008024	0.370431	0.285884	0.072511	0.252600

TABLE 9 Calculated Coal Slurry Make-Up for Tests at 160 °C

Coal Sludge Recipe										
		Sample ID	Hour Sample	%water	%Coal	%Sludge Water	%Sludge Solids	%Defoamer	%ALS	Calculated Solids
160 °C	200%	080604-MV-08	2	16.00%	64.20%	16.00%	3.80%	0.00%	0.00%	0.68000
		080604-JH-09	4	16.00%	64.28%	16.00%	3.72%	0.00%	0.00%	0.68000
		080604-JH-10	6	16.00%	63.62%	16.00%	4.38%	0.00%	0.00%	0.68000
		080704-JH-01	8	16.00%	64.19%	16.00%	3.81%	0.00%	0.00%	0.68000
	100%	080604-NB-05	2	16.00%	65.02%	16.00%	2.98%	0.00%	0.00%	0.68000
		080604-NB-05-Redo	2	16.00%	65.02%	16.00%	2.98%	0.00%	0.00%	0.68000
		080604-NB-06	4	16.00%	65.58%	16.00%	2.42%	0.00%	0.00%	0.68000
		080604-NB-06-Redo	4	16.00%	65.58%	16.00%	2.42%	0.00%	0.00%	0.68000
		080604-NB-07	6	16.00%	65.41%	16.00%	2.59%	0.00%	0.00%	0.68000
		080604-NB-07-Redo-25g	6	14.00%	65.41%	18.00%	4.59%	2.00%	0.00%	0.68000
		080604-NB-07-Redo-2.5 g	6	15.80%	65.41%	16.20%	2.79%	0.20%	0.00%	0.68000
	75%	080604-JH-02	2	16.00%	65.30%	16.00%	2.70%	0.00%	0.00%	0.68000
		080604-JH-03	4	16.00%	65.59%	16.00%	2.41%	0.00%	0.00%	0.68000
		080604-JH-03-Redo	4	14.00%	65.59%	18.00%	4.41%	2.00%	0.00%	0.68000
		080604-JH-04	6	16.00%	65.49%	16.00%	2.51%	0.00%	0.00%	0.68000
	50%	080504-JH-07	4	16.00%	65.74%	16.00%	2.26%	0.00%	0.00%	0.68000
		080504-MV-06	2	16.00%	65.55%	16.00%	2.45%	0.00%	0.00%	0.68000
		080504-JH-08	6	16.00%	65.55%	16.00%	2.45%	0.00%	0.00%	0.68000
		080604-JH-01	8	16.00%	65.61%	16.00%	2.39%	0.00%	0.00%	0.68000

TABLE 10 Percent Solids in Coal Slurry Samples for Tests at 160 °C

Weight Percent of SlurryTotal Solids								
		Sample ID	Hour Sample	wt%1	wt%2	wt%3	slurry wt%avg	slurry wt%avg Stand. Dev
160 °C	200%	080604-MV-08	2	67.86	67.99	67.77	67.87	0.1106
		080604-JH-09	4	67.66	67.69	67.77	67.71	0.05690
		080604-JH-10	6	67.49	67.64	67.58	67.57	0.07550
		080704-JH-01	8	67.64	67.84	67.69	67.72	0.1041
	100%	080604-NB-05	2	67.99	67.62	67.94	67.85	0.2007
		080604-NB-05-Redo	2	67.68	67.70	67.78	67.72	0.05290
		080604-NB-06	4	67.15	66.83	66.70	66.89	0.2316
		080604-NB-06-Redo	4	80.05	68.62	68.62	72.43	6.5991
		080604-NB-07	6	67.49	67.64	67.58	67.57	0.07550
		080604-NB-07-Redo-25g	6	68.33	68.36	68.41	68.37	0.04040
		080604-NB-07-Redo-2.5 g	6	67.51	67.31	67.01	67.28	0.2517
	75%	080604-JH-02	2	64.26	64.42	64.41	64.36	0.08960
		080604-JH-03	4	67.61	71.22	67.55	68.79	2.1018
		080604-JH-03-Redo	4	69.20	69.07	68.87	69.05	0.1662
		080604-JH-04	6	68.54	68.13	68.51	68.39	0.2285
	50%	080504-JH-07	4	69.30	69.41	68.68	69.13	0.3936
		080504-MV-06	2	69.46	69.18	68.24	68.96	0.6391
		080504-JH-08	6	72.52	72.14	71.98	72.21	0.2774
		080604-JH-01	8	67.64	67.84	67.69	67.72	0.1041

TABLE 11 Results of Coal Slurry Test, pH for Samples at 160 ^{o}C

pH							
		Sample ID	Hour Sample	pH	pOH	Hydrogen Ion Concentration, molL^{-1}	Hydroxyl Ion Concentration, molL^{-1}
160 ^{o}C	200%	080604-MV-08	2	13.46	0.54	3.47E-14	0.288403
		080604-JH-09	4	13.41	0.59	3.89E-14	0.25704
		080604-JH-10	6	13.5	0.5	3.16E-14	0.316228
		080704-JH-01	8	13.41	0.59	3.89E-14	0.25704
	100%	080604-NB-05	2	13.21	0.79	6.17E-14	0.162181
		080604-NB-05-Redo	2	13.17	0.83	6.76E-14	0.147911
		080604-NB-06	4	13.01	0.99	9.77E-14	0.102329
		080604-NB-06-Redo	4	13.23	13.23	5.89E-14	0.169824
		080604-NB-07	6	13.5	0.5	3.16E-14	0.316228
		080604-NB-07-Redo-25g	6	12.79	1.21	1.62E-13	0.06166
		080604-NB-07-Redo-2.5 g	6	12.97	1.03	1.07E-13	0.093325
	75%	080604-JH-02	2	11.3	2.7	5.01E-12	0.001995
		080604-JH-03	4	11.54	2.46	2.88E-12	0.003467
		080604-JH-03-Redo	4	12.69	1.31	2.04E-13	0.048978
		080604-JH-04	6	10.29	3.71	5.12E-11	0.000195
	50%	080504-JH-07	4	7.87	6.13	1.35E-08	7.41E-07
		080504-MV-06	2	7.7	6.3	2E-08	5.01E-07
		080504-JH-08	6	8.43	5.57	3.72E-09	2.69E-06
		080604-JH-01	8	13.41	0.59	3.89E-14	0.25704

TABLE 12 Results of Coal Slurry Test, Viscosities of Samples from Tests at 160 °C

Viscosity											
		Sample ID	Hour Sample	Initial Viscosity	Initial Torque	Initial RPM	Final Viscosity	Final torque	Final RPM	Spindle	comment
160 °C	200%	080604-MV-08	2	9600	72	3	8120	60.9	3	2	
		080604-JH-09	4	8800	66	3	6667	50	3	2	
		080604-JH-10	6	12667	95	3	8533	64	3	2	foamy
		080704-JH-01	8	9880	74	3	8800	66	3	2	foamy
	100%	080604-NB-05	2	53333	80	0.6				2	foamy
		080604-NB-05-Redo	2	8413	63.1	3	7360	55.2	3	2	foamy
		080604-NB-06	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	foamy
		080604-NB-06-Redo	4	19147	71.8	1.5	16137	60.5	1.5	2	
		080604-NB-07	6	12667	95	3	8533	64	3	2	foamy
		080604-NB-07-Redo-25g	6	7453	55.9	3	6547	49.1	3	2	
		080604-NB-07-Redo-2.5 g	6	7400	55	3	6200	46.5	3	2	foamy
	75%	080604-JH-02	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	foamy
		080604-JH-03	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	foamy
		080604-JH-03-Redo	4	22100	84	1.5	19867	74.5	1.5	2	
		080604-JH-04	6	23093	86.6	1.5	16533	62	1.5	2	
	50%	080504-JH-07	4	17787	66.7	1.5	16293	61.1	1.5	2	
		080504-MV-06	2	22400	88	1.5	19307	72.4	1.5	2	
		080504-JH-08	6	104000	77.9	0.3	118000	0.3	0.3	2	
		080604-JH-01	8	9880	74	3	8800	66	3	2	

TABLE 13 Coal Slurry Results, Raw Particle Size Distribution for Tests at 160 °C

Particle Size Distribution								
		Sample ID	Hour Sample	20	40	100	200	325
160 °C	200%	080604-MV-08	2	457.34	411.10	403.99	352.06	335.19
		080604-JH-09	4	390.76	362.36	359.05	339.24	319.63
		080604-JH-10	6	456.76	409.06	394.76	354.51	337.94
		080704-JH-01	8	390.71	362.87	358.14	338.95	320.37
	100%	080604-NB-05	2	457.19	410.51	413.18	346.59	335.13
		080604-NB-05-Redo	2	458.24	411.20	398.93	347.15	336.15
		080604-NB-06	4	390.83	362.41	366.59	339.87	318.45
		080604-NB-06-Redo	4	456.85	409.03	402.66	338.47	344.60
		080604-NB-07	6	456.76	409.06	394.76	354.51	337.94
		080604-NB-07-Redo-25g	6	456.97	414.65	400.67	346.81	334.24
		080604-NB-07-Redo-2.5 g	6	390.84	362.31	356.76	337.48	318.16
	75%	080604-JH-02	2	457.80	413.37	399.34	343.93	334.59
		080604-JH-03	4	456.96	410.31	402.82	346.30	334.32
		080604-JH-03-Redo	4	458.41	415.60	397.17	346.92	335.92
		080604-JH-04	6	457.02	409.45	400.21	351.17	334.76
	50%	080504-JH-07	4	391.15	363.32	356.78	338.66	316.25
		080504-MV-06	2	390.58	363.89	355.71	336.58	317.36
		080504-JH-08	6	459.27	419.88	396.84	347.35	333.89
		080604-JH-01	8	390.71	362.87	358.14	338.95	320.37

TABLE 14 Sieves Used for Specific Coal Slurry Tests at 160 ^{o}C

Particle Size Distribution									
		Sample ID	Hour Sample	Set	standard weight				
160 °C	200%	080604-MV-08	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-JH-09	4	Seive 2	390.22	361.28	331.32	325.24	312.32
		080604-JH-10	6	Seive 1	456.39	408.51	375.29	335.49	331.27
		080704-JH-01	8	Seive 2	390.22	361.28	331.32	325.24	312.32
	100%	080604-NB-05	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-NB-05-Redo	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-NB-06	4	Seive 2	390.22	361.28	331.32	325.24	312.32
		080604-NB-06-Redo	4	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-NB-07	6	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-NB-07-Redo-25g	6	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-NB-07-Redo-2.5 g	6	Seive 2	390.22	361.28	331.32	325.24	312.32
	75%	080604-JH-02	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-JH-03	4	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-JH-03-Redo	4	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-JH-04	6	Seive 1	456.39	408.51	375.29	335.49	331.27
	50%	080504-JH-07	4	Seive 2	390.22	361.28	331.32	325.24	312.32
		080504-MV-06	2	Seive 2	390.22	361.28	331.32	325.24	312.32
		080504-JH-08	6	Seive 1	456.39	408.51	375.29	335.49	331.27
		080604-JH-01	8	Seive 2	390.22	361.28	331.32	325.24	312.32

TABLE 15 Coal Slurry Results, Difference Particle Size Distribution for Tests at 160 °C

Particle Size Distribution											
		Sample ID	Hour Sample	Difference					Total	Total Solids of Sample	difference
160 °C	200%	080604-MV-08	2	0.95	2.59	28.70	16.57	3.92	52.73	67.87	15.14
		080604-JH-09	4	0.54	1.08	27.73	14.00	7.31	50.66	67.71	17.05
		080604-JH-10	6	0.37	0.55	19.47	19.02	6.67	46.08	67.57	21.49
		080704-JH-01	8	0.49	1.59	26.82	13.71	8.05	50.66	67.72	17.06
	100%	080604-NB-05	2	0.80	2.00	37.89	11.10	3.86	55.65	67.85	12.20
		080604-NB-05-Redo	2	1.85	2.69	23.64	11.66	4.88	44.72	67.72	23.00
		080604-NB-06	4	0.61	1.13	35.27	14.63	6.13	57.77	66.89	9.12
		080604-NB-06-Redo	4	0.46	0.52	27.37	2.98	13.33	44.66	72.43	27.77
		080604-NB-07	6	0.37	0.55	19.47	19.02	6.67	46.08	67.57	21.49
		080604-NB-07-Redo-25g	6	0.58	6.14	25.38	11.32	2.97	46.39	68.37	21.98
		080604-NB-07-Redo-2.5 g	6	0.62	1.03	25.44	12.24	5.84	45.17	67.28	22.11
	75%	080604-JH-02	2	1.41	4.86	24.05	8.44	3.32	42.08	64.36	22.28
		080604-JH-03	4	0.57	1.80	27.53	10.81	3.05	43.76	68.79	25.03
		080604-JH-03-Redo	4	2.02	7.09	21.88	11.43	4.65	47.07	69.05	21.98
		080604-JH-04	6	0.63	0.94	24.92	15.68	3.49	45.66	68.39	22.73
	50%	080504-JH-07	4	0.93	2.04	25.46	13.42	3.93	45.78	69.13	23.35
		080504-MV-06	2	0.36	2.61	24.39	11.34	5.04	43.74	68.96	25.22
		080504-JH-08	6	2.88	11.37	21.55	11.86	2.62	50.28	72.21	21.93
		080604-JH-01	8	0.49	1.59	26.82	13.71	8.05	50.66	67.72	17.06

TABLE 16 Coal slurry results, normalized particle size distribution for tests at 160 °C

Particle Size Distribution									
		Sample ID	Hour Sample	Normalized					>325 mesh size
160 °C	200%	080604-MV-08	2	0.01400	0.03816	0.42285	0.24413	0.05775	0.22311
		080604-JH-09	4	0.00798	0.01595	0.40956	0.20677	0.10797	0.25177
		080604-JH-10	6	0.00548	0.00814	0.28815	0.28149	0.09871	0.31804
		080704-JH-01	8	0.00724	0.02348	0.39602	0.20244	0.11887	0.25196
	100%	080604-NB-05	2	0.01179	0.02948	0.55844	0.16360	0.05689	0.17981
		080604-NB-05-Redo	2	0.02732	0.03972	0.34908	0.17218	0.07206	0.33963
		080604-NB-06	4	0.00912	0.01689	0.52726	0.21871	0.09164	0.13639
		080604-NB-06-Redo	4	0.00635	0.00718	0.37788	0.04114	0.18404	0.38340
		080604-NB-07	6	0.00548	0.00814	0.28815	0.28149	0.09871	0.31804
		080604-NB-07-Redo-25g	6	0.00848	0.08981	0.37123	0.16558	0.04344	0.32145
		080604-NB-07-Redo-2.5 g	6	0.00922	0.01531	0.37814	0.18194	0.08681	0.32859
	75%	080604-JH-02	2	0.02191	0.07551	0.37366	0.13113	0.05158	0.34621
		080604-JH-03	4	0.00829	0.02617	0.40018	0.15714	0.04434	0.36389
		080604-JH-03-Redo	4	0.02926	0.10268	0.31689	0.16554	0.06735	0.31829
		080604-JH-04	6	0.00921	0.01374	0.36436	0.22926	0.05103	0.33239
	50%	080504-JH-07	4	0.01345	0.02951	0.36829	0.19413	0.05685	0.33777
		080504-MV-06	2	0.00522	0.03785	0.35368	0.16444	0.07309	0.36572
		080504-JH-08	6	0.03988	0.15745	0.29842	0.16424	0.03628	0.30373
		080604-JH-01	8	0.00724	0.02348	0.39602	0.20244	0.11887	0.25196

TABLE 17 Calculated Coal Slurry Make-Up for Tests at 150 °C

Coal Sludge Recipe									
		Sample ID	Hour Sample	%Water	%Coal	%Sludge Water	%Sludge Solids	%Defoamer	%ALS
150 °C	200%	072604-NB-01	2	16.00%	63.89%	16.00%	4.11%	0.00%	0.00%
		072604-JH-02	4	16.00%	65.22%	16.00%	2.78%	0.00%	0.00%
	100%	072704-NB-01	2	16.00%	64.74%	16.00%	3.26%	0.00%	0.00%
		072704-JH-02-CORRECTED	4	16.00%	65.00%	16.00%	3.00%	0.00%	0.00%
		072704-JH-02-ACTUAL		16.00%	65.09%	16.00%	2.91%	0.00%	0.00%
	75%	072804-NB-01	2	16.00%	66.15%	16.00%	1.85%	0.00%	0.00%
	50%	080504-NB-04	2	16.00%	65.98%	16.00%	2.02%	0.00%	0.00%
		080504-NB-05	4	16.00%	65.52%	16.00%	2.48%	0.00%	0.00%

TABLE 18 Percent Solids in Coal Slurry Samples for Tests at 150 °C

Weight Percent of Slurry Total Solids								
		Sample ID	Hour Sample	wt%1	wt%2	wt%3	slurry wt%avg	slurry wt%avg Stand. Dev
150 °C	200%	072604-NB-01	2	69.41	69.33	69.25	69.33	0.0800
		072604-JH-02	4	68.31	68.20	67.72	68.08	0.3137
	100%	072704-NB-01	2	71.74	71.40	71.30	71.48	0.2307
		072704-JH-02-CORRECTED	4	69.12	69.22	68.57	68.97	0.3500
		072704-JH-02-ACTUAL		69.12	69.22	68.57	68.97	0.3500
	75%	072804-NB-01	2	68.98	77.42	68.94	71.78	4.8844
	50%	080504-NB-04	2	68.85	67.88	67.43	68.05	0.7257
		080504-NB-05	4	68.46	68.56	68.64	68.55	0.0902

TABLE 19 Results of Coal Slurry Test, Viscosities of Samples from Tests at 150 °C

Viscosity										
		Sample ID	Hour Sample	Initial Viscosity	Initial Torque	Initial RPM	Final Viscosity	Final torque	Final RPM	Spindle
150 °C	200%	072604-NB-01	2	11013	82.6	3	8200	61.5	3	2
		072604-JH-02	4	8800	65.9	3	7880	59	3	2
	100%	072704-NB-01	2	18181	65.3	1.5	11200	42	1.5	2
		072704-JH-02-CORRECTED	4	20907	78.4	1.5	17413	65.3	1.5	2
		072704-JH-02-ACTUAL		20907	78.4	1.5	17413	65.3	1.5	2
	75%	072804-NB-01	2	9920	74.4	3	7173	53.8	3	2
	50%	080504-NB-04	2	82400	62.8	0.3	74400	55.8	0.3	2
		080504-NB-05	4	122000	91.6	0.3	109000	81.8	0.3	2

TABLE 20 Results of Coal Slurry Test, pH for Samples at 150 °C

pH							
		Sample ID	Hour Sample	pH	pOH	hydrogen Ion Concentration	Hydroxyl Ion Concentration
150 °C	200%	072604-NB-01	2	14.08	-0.08	8.32E-15	1.202264
		072604-JH-02	4	13.92	0.08	1.2E-14	0.831764
	100%	072704-NB-01	2	13.56	0.44	2.75E-14	0.363078
		072704-JH-02-CORRECTED	4	13.51	0.49	3.09E-14	0.323594
		072704-JH-02-ACTUAL		13.51	0.49	3.09E-14	0.323594
	75%	072804-NB-01	2	13.18	0.82	6.61E-14	0.151356
	50%	080504-NB-04	2	10.18	3.82	6.61E-11	0.000151
		080504-NB-05	4	10.7	3.3	2E-11	0.000501

TABLE 21 Coal Slurry Results, Raw Particle Size Distribution for Tests at 150 °C

Particle Size Distribution								
		Sample ID	Hour Sample	20	40	100	200	325
150 °C	200%	072604-NB-01	2	457.08	410.98	397.91	347.39	334.46
		072604-JH-02	4	390.72	362.93	354.97	336.82	318.42
	100%	072704-NB-01	2	466.29	410.92	387.97	346.27	334.93
		072704-JH-02-CORRECTED	4	390.55	362.47	351.33	339.96	318.21
		072704-JH-02-ACTUAL						
	75%	072804-NB-01	2	459.28	410.19	394.52	350.44	333.67
	50%	080504-NB-04	2	396.45	365.10	351.48	335.48	317.77
		080504-NB-05	4	390.81	362.45	355.80	336.76	317.92

TABLE 22 Sieves Used for Specific Coal Slurry Tests at 150 ^{o}C

Particle Size Distribution									
		Sample ID	Hour Sample	Set	standard weight				
150 °C	200%	072604-NB-01	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		072604-JH-02	4	Seive 2	390.22	361.28	331.32	325.24	312.32
	100%	072704-NB-01	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		072704-JH-02-CORRECTED	4	Seive 2	390.22	361.28	331.32	325.24	312.32
		072704-JH-02-ACTUAL							
	75%	072804-NB-01	2	Seive 1	456.39	408.51	375.29	335.49	331.27
	50%	080504-NB-04	2	Seive 2	390.22	361.28	331.32	325.24	312.32
		080504-NB-05	4	Seive 2	390.22	361.28	331.32	325.24	312.32

TABLE 23 Coal Slurry Results, Difference Particle Size Distribution for Tests at 150 °C

Particle Size Distribution											
		Sample ID	Hour Sample	difference					Total		difference
150 °C	200%	072604-NB-01	2	0.69	2.47	22.62	11.90	3.19	40.87	69.33	28.46
		072604-JH-02	4	0.50	1.65	23.65	11.58	6.10	43.48	68.08	24.60
	100%	072704-NB-01	2	9.90	2.41	12.68	10.78	3.66	39.43	71.48	32.05
		072704-JH-02-CORRECTED	4	0.33	1.19	20.01	14.72	5.89	42.14	68.97	26.83
		072704-JH-02-ACTUAL		0.00	0.00	0.00	0.00	0.00	0.00	68.97	68.97
	75%	072804-NB-01	2	2.89	1.68	19.23	14.95	2.40	41.15	71.78	30.63
	50%	080504-NB-04	2	6.23	3.82	20.16	10.24	5.45	45.90	68.05	22.15
		080504-NB-05	4	0.59	1.17	24.48	11.52	5.60	43.36	68.55	25.19

TABLE 24 Coal Slurry Results, Normalized Particle Size Distribution for Tests at 150 °C

Particle Size Distribution									
		Sample ID	Hour Sample	Normalized					>325 mesh size
150 °C	200%	072604-NB-01	2	0.0100	0.0356	0.3263	0.1716	0.0460	0.4105
		072604-JH-02	4	0.0073	0.0242	0.3474	0.1701	0.0896	0.3613
	100%	072704-NB-01	2	0.1385	0.0337	0.1774	0.1508	0.0512	0.4484
		072704-JH-02-CORRECTED	4	0.0048	0.0173	0.2901	0.2134	0.0854	0.3890
		072704-JH-02-ACTUAL		0.0000	0.0000	0.0000	0.0000	0.0000	1.0000
	75%	072804-NB-01	2	0.0403	0.0234	0.2679	0.2083	0.0334	0.4267
	50%	080504-NB-04	2	0.0915	0.0561	0.2962	0.1505	0.0801	0.3255
		080504-NB-05	4	0.0086	0.0171	0.3571	0.1680	0.0817	0.3675

TABLE 25 Calculated Coal Slurry Make-Up for Tests at 140 °C

Coal Grind Calculated Distribution										
		Sample ID	Hour Sample	Calculated Solids	%Water	%Coal	%Sludge Water	%Sludge Solids	%Defoamer	%ALS
140 °C	200%	080304-JH-01	2	0.68000	16.00%	65.41%	16.00%	2.59%	0.00%	0.00%
		080304-JH-02	4	0.68000	16.00%	65.06%	16.00%	2.94%	0.00%	0.00%
		080304-JH-03	6	0.68000	16.00%	64.90%	16.00%	3.10%	0.00%	0.00%
	100%	080405-NB-01	2	0.68000	16.00%	65.58%	16.00%	2.42%	0.00%	0.00%
		080405-NB-02	4	0.68000	16.00%	65.35%	16.00%	2.65%	0.00%	0.00%
		080405-NB-03	6	0.68000	16.00%	65.25%	16.00%	2.75%	0.00%	0.00%
	75%	080404-JH-04	2	0.68000	16.00%	65.41%	16.00%	2.59%	0.00%	0.00%
		080404-JH-05	4	0.68000	16.00%	65.47%	16.00%	2.53%	0.00%	0.00%
	50%	080504-JH-01	2	0.68000	16.00%	65.32%	16.00%	2.68%	0.00%	0.00%
		080504-JH-02	4	0.68000	16.00%	65.67%	16.00%	2.33%	0.00%	0.00%

TABLE 26 Percent Solids in Coal Slurry Samples for Tests at 140 °C

Weight Percent of Slurry Total Solids								
		Sample ID	Hour Sample	wt%1	wt%2	wt%3	Slurry wt%avg	Slurry wt%avg Stand. Dev
140 °C	200%	080304-JH-01	2	69.31	69.15	69.10	69.19	0.1097
		080304-JH-02	4	69.73	69.71	69.60	69.68	0.07000
		080304-JH-03	6	69.23	69.55	66.02	68.27	1.952
	100%	080405-NB-01	2	68.74	70.12	69.82	69.56	0.7258
		080405-NB-02	4	66.22	66.03	66.76	66.34	0.3787
		080405-NB-03	6	68.89	69.90	70.91	69.90	1.010
	75%	080404-JH-04	2	63.45	64.38	65.84	64.56	1.205
		080404-JH-05	4	68.72	69.79	69.24	69.25	0.5351
	50%	080504-JH-01	2	68.46	68.34	67.62	68.14	0.4543
		080504-JH-02	4	67.43	67.50	67.28	67.40	0.1124

TABLE 27 Results of Coal Slurry Test, pH for Samples at 140 ^{o}C

pH							
		Sample ID	Hour Sample	pH	pOH	Hydrogen Ion Concentration, molL^{-1}	Hydroxyl Ion Concentration, molL^{-1}
140 ^{o}C	200%	080304-JH-01	2	13.56	0.440	2.754E-14	0.3631
		080304-JH-02	4	13.50	0.500	3.162E-14	0.3162
		080304-JH-03	6	13.68	0.320	2.089E-14	0.4786
	100%	080405-NB-01	2	13.17	0.830	6.761E-14	0.1479
		080405-NB-02	4	13.01	0.990	9.772E-14	0.1023
		080405-NB-03	6	13.09	0.910	8.128E-14	0.1230
	75%	080404-JH-04	2	13.19	0.810	6.457E-14	0.1549
		080404-JH-05	4	12.50	1.500	3.162E-13	0.0316
	50%	080504-JH-01	2	12.31	1.690	4.898E-13	0.0204
		080504-JH-02	4	10.78	3.220	1.660E-11	0.0006

TABLE 28 Results of Coal Slurry Test, Viscosities of Samples from Tests at 140 °C

Viscosity											
		Sample ID	Hour Sample	Initial Viscosity	Initial Torque	Initial RPM	Final Viscosity	Final torque	Final RPM	Spindle	comments
140 °C	200%	080304-JH-01	2	20240	76	1.5	15013	56.3	1.5	2	
		080304-JH-02	4	21280	79.8	1.5	14960	56.1	1.5	2	foamy
		080304-JH-03	6	90267	67.7	0.3	72000	54	0.3	2	foamy
	100%	080405-NB-01	2	75467	56.6	0.3	77333	58	0.3	2	foamy
		080405-NB-02	4	129000	96.8	0.3	131000	98.5	0.3	2	foamy
		080405-NB-03	6	103000	77.5	0.3	88933	66.8	0.3	2	foamy
	75%	080404-JH-04	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	foamy
		080404-JH-05	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	foamy
	50%	080504-JH-01	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	foamy
		080504-JH-02	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	foamy

TABLE 29 Coal Slurry Results, Raw Particle Size Distribution for Tests at 140 °C

Mass of Trays with Solids for Particle Size Distribution								
		Sample ID	Hour Sample	20	40	100	200	325
140 °C	200%	080304-JH-01	2	457.02	411.99	398.04	346.75	333.09
		080304-JH-02	4	391.63	363.38	354.60	339.48	318.79
		080304-JH-03	6	465.57	412.45	392.38	344.54	333.92
	100%	080405-NB-01	2	402.25	369.63	344.98	332.80	316.53
		080405-NB-02	4	466.89	415.88	389.04	337.01	337.93
		080405-NB-03	6	414.96	367.62	341.46	332.77	316.85
	75%	080404-JH-04	2	469.27	414.19	387.98	342.87	334.56
		080404-JH-05	4	402.07	369.25	346.46	333.82	315.84
	50%	080504-JH-01	2	468.83	417.57	389.30	341.82	332.03
		080504-JH-02	4	390.72	363.57	356.11	336.69	316.31

TABLE 30 Sieves Used for Specific Coal Slurry Tests at 140 ^{o}C

Seive Weights Used for Particle Size Distribution									
					standard weight				
		Sample ID	Hour Sample	Set	20	40	100	200	325
140 °C	200%	080304-JH-01	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		080304-JH-02	4	Seive 2	390.22	361.28	331.32	325.24	312.32
		080304-JH-03	6	Seive 1	456.39	408.51	375.29	335.49	331.27
	100%	080405-NB-01	2	Seive 2	390.22	361.28	331.32	325.24	312.32
		080405-NB-02	4	Seive 1	456.39	408.51	375.29	335.49	331.27
		080405-NB-03	6	Seive 2	390.22	361.28	331.32	325.24	312.32
	75%	080404-JH-04	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		080404-JH-05	4	Seive 2	390.22	361.28	331.32	325.24	312.32
	50%	080504-JH-01	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		080504-JH-02	4	Seive 2	390.22	361.28	331.32	325.24	312.32

TABLE 31 Coal Slurry Results, Difference Particle Size Distribution for Tests at 140 °C

Difference Particle Size Distribution											
				Difference					Total	Total Solids	difference
		Sample ID	Hour Sample	20	40	100	200	325			>325
140 °C	200%	080304-JH-01	2	0.63	3.48	22.75	11.26	1.82	39.94	69.19	29.25
		080304-JH-02	4	1.41	2.10	23.28	14.24	6.47	47.50	69.68	22.18
		080304-JH-03	6	9.18	3.94	17.09	9.05	2.65	41.91	68.27	26.36
	100%	080405-NB-01	2	12.03	8.35	13.66	7.56	4.21	45.81	69.56	23.75
		080405-NB-02	4	10.50	7.37	13.75	1.52	6.66	39.80	66.34	26.54
		080405-NB-03	6	24.74	6.34	10.14	7.53	4.53	53.28	69.90	16.62
	75%	080404-JH-04	2	12.88	5.68	12.69	7.38	3.29	41.92	64.56	22.64
		080404-JH-05	4	11.85	7.97	15.14	8.58	3.52	47.06	69.25	22.19
	50%	080504-JH-01	2	12.44	9.06	14.01	6.33	0.76	42.60	68.14	25.54
		080504-JH-02	4	0.50	2.29	24.79	11.45	3.99	43.02	67.40	24.38

TABLE 32 Coal Slurry Results, Normalized Particle Size Distribution for Tests at 140 °C

Normalized Particle Size Distribution									
				Normalized					
		Sample ID	Hour Sample	20	40	100	200	325	>325 mesh size
140 °C	200%	080304-JH-01	2	0.009106	0.050299	0.328821	0.162748	0.026306	0.422721
		080304-JH-02	4	0.020235	0.030138	0.334099	0.204363	0.092853	0.318312
		080304-JH-03	6	0.134473	0.057715	0.250342	0.132568	0.038818	0.386084
	100%	080405-NB-01	2	0.172944	0.120040	0.196377	0.108683	0.060523	0.341432
		080405-NB-02	4	0.158284	0.111100	0.207276	0.022913	0.100397	0.400030
		080405-NB-03	6	0.353934	0.090701	0.145064	0.107725	0.064807	0.237768
	75%	080404-JH-04	2	0.199515	0.087985	0.196571	0.114318	0.050963	0.649352
		080404-JH-05	4	0.171119	0.115090	0.218628	0.123899	0.050830	0.320433
	50%	080504-JH-01	2	0.182565	0.132962	0.205606	0.092897	0.011154	0.374817
		080504-JH-02	4	0.007418	0.033975	0.367786	0.169873	0.059196	0.361753

TABLE 33 Calculated Coal Slurry Make-Up for Tests at 160 ^{o}C H_2SO_4

Coal Sludge Recipe										
		Sample	Hour	%water	%Coal	%Sludge Water	%Sludge Solids	%Defoamer	%ALS	Calculated Solids
160 °C Sulfuric Acid	100%	081104-JH-01	2	15.97%	65.73%	16.00%	2.14%	0.00%	0.00%	68.00%
		081104-JH-01-Redo	2	14.17%	65.73%	17.94%	3.97%	1.01%	0.00%	68.55%
		081104-JH-02	4	15.97%	65.88%	16.00%	1.99%	0.00%	0.00%	68.00%
		081104-JH-03	6	15.97%	66.09%	16.00%	1.78%	0.00%	0.00%	68.00%
160 °C Sulfuric Acid	50%	081204-NB-01	2	15.97%	65.95%	16.00%	1.92%	0.00%	0.00%	68.00%
		081204-NB-02	4	15.97%	66.00%	16.00%	1.87%	0.00%	0.00%	68.00%

TABLE 34 Percent Solids in Coal Slurry Samples for Tests at 160 ^{o}C H_2SO_4

Weight Percent Total Solids								
		Sample	Hour	wt%1	wt%2	wt%3	slurry wt% avg	slurry wt% Stand. Dev
160 °C Sulfuric Acid	100%	081104-JH-01	2	71.45	71.48	71.19	71.37	0.1595
		081104-JH-01-Redo	2	70.94		70.46	70.70	0.3394
		081104-JH-02	4	69.56	69.90	70.14	69.87	0.2914
		081104-JH-03	6	69.88	69.92	69.51	69.77	0.2261
160 °C Sulfuric Acid	50%	081204-NB-01	2	73.33	72.88	72.47	71.96	0.2511
		081204-NB-02	4	72.22	71.93	71.72	72.89	0.4302

TABLE 35 Results of Coal Slurry Test, pH for Samples at 160 ^{o}C H_2SO_4

pH							
		Sample	Hour	pH	pOH	hydrogen Ion Concentration	Hydroxyl Ion Concentration
160 °C Sulfuric Acid	100%	081104-JH-01	2	0.95	13.05	0.112202	8.91E-14
		081104-JH-01-Redo	2	0.95	13.05	0.112202	8.91E-14
		081104-JH-02	4	0.21	13.79	0.616595	1.62E-14
		081104-JH-03	6	0.36	13.64	0.436516	2.29E-14
160 °C Sulfuric Acid	50%	081204-NB-01	2	1.12	12.88	0.075858	1.32E-13
		081204-NB-02	4	0.98	13.02	0.104713	9.55E-14

TABLE 36 Results of Coal Slurry Test, Viscosities of Samples from Tests at 160 ^{o}C H_2SO_4

		Sample	Hour	Initial Viscosity	Initial Torque	Initial RPM	Final Viscosity	Final Torque	Final RPM	Spindle	Comment
160 ^{o}C Sulfuric Acid	100%	081104-JH-01	2	3293	24.7	3	3677	27.5	3	2	
		081104-JH-01-Redo	2	7747	58.1	3	6627	49.7	3	2	
		081104-JH-02	4	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Very Viscous
		081104-JH-03	6	11400	85.5	3	11527	87.2	3	2	
160 ^{o}C Sulfuric Acid	50%	081204-NB-01	2	N/A	N/A	N/A	N/A	N/A	N/A	N/A	Foamy
		081204-NB-02	4	108000	80.8	0.3	127000	95	0.3	2	Very Viscous

TABLE 37 Coal Slurry Results, Raw Particle Size Distribution for Tests at 160 ^{o}C H_2SO_4

Particle Size Distribution								
		Sample	Hour	20	40	100	200	325
160 °C Sulfuric Acid	100%	081104-JH-01	2	470.87	418.34	384.48	341.65	332.58
		081104-JH-01-Redo	2	397.47	368.77	348.87	337.34	316.8
		081104-JH-02	4	467.77	418.95	386.16	342.26	333.38
		081104-JH-03	6	391.53	369.13	350.02	335.18	317.86
160 °C Sulfuric Acid	50%	081204-NB-01	2	504.61	416.24	379.96	337.38	331.5
		081204-NB-02	4	433.39	370.16	337	328.58	315.25

TABLE 38 Sieves Used for Specific Coal Slurry Tests at 160 ^{o}C H_2SO_4

Particle Size Distribution									
					standard weight				
		Sample	Hour	Set	20	40	100	200	325
160 °C Sulfuric Acid	100%	081104-JH-01	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		081104-JH-01-Redo	2	Seive 2	390.22	361.28	331.32	325.24	312.32
		081104-JH-02	4	Seive 1	456.39	408.51	375.29	335.49	331.27
		081104-JH-03	6	Seive 2	390.22	361.28	331.32	325.24	312.32
160 °C Sulfuric Acid	50%	081204-NB-01	2	Seive 1	456.39	408.51	375.29	335.49	331.27
		081204-NB-02	4	Seive 2	390.22	361.28	331.32	325.24	312.32

TABLE 39 Coal Slurry Results, Difference Particle Size Distribution for Tests at 160 ^{o}C H_2SO_4

Particle Size Distribution											
				Difference							
		Sample	Hour	20	40	100	200	325	Total	Total Solids in Sample	difference
160 °C Sulfuric Acid	100%	081104-JH-01	2	14.48	9.83	9.19	6.16	1.31	40.97	71.37	30.40
		081104-JH-01-Redo	2	7.25	7.49	17.55	12.10	4.48	48.87	70.70	21.83
		081104-JH-02	4	11.38	10.44	10.87	6.77	2.11	41.57	69.87	28.30
		081104-JH-03	6	1.31	7.85	18.70	9.94	5.54	43.34	69.77	26.43
160 °C Sulfuric Acid	50%	081204-NB-01	2	48.22	7.73	4.67	1.89	0.23	62.74	71.96	9.22
		081204-NB-02	4	43.17	8.88	5.68	3.34	2.93	64.00	72.89	8.89

TABLE 40 Coal Slurry Results, Normalized Particle Size Distribution for Tests at 160 ^{o}C H_2SO_4

Particle Size Distribution									
				Normalized					
		Sample	Hour	20	40	100	200	325	>325 mesh size
160 ^{o}C Sulfuric Acid	100%	081104-JH-01	2	0.2029	0.1377	0.1288	0.0863	0.0184	0.4260
		081104-JH-01-Redo	2	0.1025	0.1059	0.2482	0.1711	0.0634	0.3088
		081104-JH-02	4	0.1629	0.1494	0.1556	0.0969	0.0302	0.4050
		081104-JH-03	6	0.0188	0.1125	0.2680	0.1425	0.0794	0.3788
160 ^{o}C Sulfuric Acid	50%	081204-NB-01	2	0.6701	0.1074	0.0649	0.0263	0.0032	0.1281
		081204-NB-02	4	0.5922	0.1218	0.0779	0.0458	0.0402	0.1220

VITA

Jacob Eli Hutton was born in Concord, Massachusetts on January 24, 1977. He was raised in Littleton, MA until his junior year in high school, where he moved to Oak Ridge, TN to finish his secondary education. After graduation from Oak Ridge High School, he went on to complete a Bachelor of Science in Chemical Engineering at the University of Tennessee, May, 2002.

Jacob is currently resides in Knoxville, TN and completing his Masters of Science degree in Chemical Engineering at the University of Tennessee.